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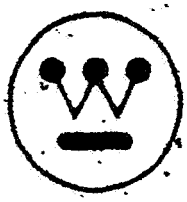
Subcontract NP-1

WANL-TME-1811
JUNE 1968



Westinghouse Astronuclear Laboratory

SUMMARY OF WESTINGHOUSE ASTRONUCLEAR LABORATORY RAW MATERIALS ACTIVITIES FOR NERVA APPLICATION

(Title Unclassified)

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This document is classified in accordance with the Joint Classification Guide Nuclear Rocket Propulsion Systems (ROVER), CG-RR-2, Topic 8.0.

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6/17/68
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Date

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FOREWORD

The Westinghouse Astronuclear Laboratory procures production quantities of raw materials for NERVA reactor fuel elements in accord with the WANL-TNR-088 interpretation of the requirements specified in NASA NPC 200-2. Extensive qualification of raw materials is performed in NERVA processes to assure conformance with these specifications. Continuing programs are necessary to provide evaluation of raw material interactions with fuel element performance, specification upgrading, supplier qualification, and supplier process control.

This report summarizes the present status of Purchasing Department Specifications used for raw material procurement. The specifications are included in Appendix C.

The experience and skills available within NERVA Fuel and Materials, Product Assurance, Corporate Purchasing Departments, and the Corporate Specification and Controls Activities are utilized to insure a comprehensive raw materials procurement program at the Astronuclear Laboratory. The result is a positive program of maintaining inclusive bidders' lists, qualification of suppliers, and assured control of incoming raw materials.

SUMMARY TABLE I

RAW MATERIAL ACCEPTANCE CRITERIA

	Maximum Allowable Impurities	Minimum Assay	Particle Size	Density	Moisture Content	Isotropy and Structure	Surface Area	Billet Cross-Sectional Area	Graphitization Temperature	Radiographic Inspection	Solids Content	Viscosity	Gel Time	pH	Particle Shape	Uranium Content	Thermal Migration Resistance	Acid Leachable Uranium	Pressure of Protective Atmosphere	X-Ray Diffraction Pattern	Insoluble In Chloroform	Alloy Composition	Flexural Strength	Elevated Temperature Creep	Thermal Expansion	Porosity
Graphite Flour	X		X	X	X	X	X	X	X																	
Carbon Black	X		X	X	X					X																
Furfuryl Alcohol Resin	X										X	X	X	X												
Maleic Anhydride	X	X																								
Fuel Beads	X		X	X											X	X	X	X								
Niobium Pentachloride	X	X	X																X							
Zirconium Tetrachloride	X	X	X																X							
Molybdenum Hexacarbonyl	X	X																		X	X					
Hydrogen Chloride	X	X																								
Carrier Gas (Argon)	X	X																								
Hydrogen	X	X																								
Methane	X	X																								
Molybdenum Braze Powder	X	X	X																							
Key Braze Powder	X		X																			X				
Carbide/Graphite Composite				X						X													X	X	X	
Carbon Powder	X		X			X																				
Niobium Carbide Powder	X	X	X	X																						X

SUMMARY TABLE II

SUPPLIER QUALIFICATION STATUS AND RAW MATERIAL COSTS

Raw Material	Supplier	Status	Raw Material Cost
Graphite Flour (PDS-30046-C)	Great Lakes Carbon Company	Accepted PDS-30046-C, order being processed for qualification.	$1.19/lb
	Speer Carbon Company	Indicates future interest as a potential supplier.	-----
	Union Carbide Corporation	Indicates future interest as a potential supplier.	-----
	Stackpole Carbon Company	No interest indicated as a supplier.	------
	Saint Mary's Carbon Company	No interest indicated as a supplier.	------
	Asbury Graphite Mills	Indicates future interest as a potential supplier to custom particle size distributions.	------
	Pure Carbon Company	No interest indicated as a supplier.	-----
	Ultra Carbon Company	No interest indicated as a supplier.	-----
	Carborundum Company	No interest indicated as a supplier.	-----
Carbon Black (PDS-30047-B)	R. T. Vanderbilt Company	Qualified	$0.06/lb

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SUMMARY TABLE II (CONTINUED)

Raw Material	Supplier	Status	Raw Material Cost
Partially Polymerized Furfuryl Alcohol Resin (PDS-30048-B)	Varcum Chemical Company	Qualified	$0.45/lb
	Durez Division of Hooker Chemical Company	Samples ordered for evaluation to AFF 3001.	----
Maleic Anhydride (PDS-51300-AB)	Fisher Scientific Company	Qualified	$3.50/lb
Pyrocarbon Coated Uranium Dicarbide Fuel Beads (PDS-30050-2-E)	Gulf-General Atomic	Qualified	$449.00/kg
	Minnesota Mining and Manufacturing	No interest indicated as a supplier.	----
	Carbon Products Division of Union Carbide Corporation	No interest indicated as a supplier.	----
	Nuclear Fuel and Materials Corporation	Indicates future interest as a potential supplier.	----
	Nuclear Fuel Services	Has not indicated status at this time.	----
Molybdenum Brazing Powder (PDS-30179-A)	Fansteel Metallurgical	Qualified	$5.25/lb
	Sylvania Electric	Qualified	----
Carbide/Graphite Composite Material (PDS-30106-E)	Carborundum Company	Qualified	$143.00/lb
	Norton Company	Qualified	$143.00/lb
	Donald W. Douglas Laboratories	Material has not met qualification requirements.	----

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SUMMARY TABLE II (CONTINUED)

Raw Material	Supplier	Status	Raw Material Cost
Carbide/Graphite Composite Material (PDS-30106-E (Cont'd.)	Supertemp Corporation	Material has not met qualification requirements.	----
	Union Carbide Corporation	Material has not met qualification requirements.	----
Carbon Powder (PDS-30106-E)	Union Carbide Corporation	Qualified	$1.00/lb
	Carborundum Company	Qualified	$1.00/lb
Niobium Carbide Powder (PDS-30104-E)	Kawecki Chemical Company	Qualified	$15.75/lb
	Wah Chang Company	Material has not met qualification requirements.	----
	Kennametal Company	Material has not met qualification requirements.	----
	Fansteel Metallurgical	Material has not met qualification requirements.	----
Niobium (Columbium) Pentachloride (PDS-30052-D)	Stauffer Chemical Company	Qualified	$4.60/lb with Y-12 supplied niobium.
	Atomergic Chemical Company	Qualified	$8.00/lb including niobium cost.
	Wah Chang Company	Order being processed for qualification.	$7.50/lb including niobium cost.

SUMMARY TABLE II (CONTINUED)

Raw Material	Supplier	Status	Raw Material Cost
Zirconium Tetrachloride (PDS-30131-1)	Wah Chang Company	Invitation for quotation being processed.	----
Molybdenum Hexacarbonyl (PDS-30095)	Climax Molybdenum	Qualified	$26.45/lb
	Pressure Chemicals Company	Order being processed for qualification.	$26.45/lb
Anhydrous Hydrogen Chloride (PDS-52217-AP)	Frontier Chemical Company	Qualified to supply the commercial grade.	$0.46/lb
	Air Products Corporation	Qualified to supply the electronic grade.	$3.05/lb
Inert Carrier Gas (Argon) (PDS-52118-BA)	Air Products Corporation	Qualified	$1.13/100 cu ft
Hydrogen (PDS-3057-F-3)	Air Products Corporation	Qualified	$1.00/100 cu ft
Methane (PDS-30130)	Matheson Company	Invitation for quotation being processed.	----
Key Brazing Powder (PDS-30177)	Brush Beryllium Corporation	Qualified	$10.00/oz

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1.0 INTRODUCTION

WNCO procures the production quantities of raw materials used by WANL to manufacture NERVA fuel elements. Raw materials are purchased to the requirements of Purchasing Department Specifications. Quality surveillance is applied in the supplier's plant, where required, and incoming material is subjected to Quality Control action at the time of delivery. Included are receiving inspection, sampling and analyses to specification requirements, and identification of containers. Appendix A includes the applicable QMP's.

Each supplier's material is qualified in NERVA processes. Engineering defines the manufacture of evaluation fuel elements, both for each new supplier and in the event of changes in the manufacturing processes of previously qualified suppliers. Evaluation hardware is subjected to the drawing and specification tests required to insure compliance with work orders specifying deliverable hardware. In the cases of graphite flour, carbon black, molybdenum hexacarbonyl, and partially polymerized furfuryl alcohol resin, qualification is performed on a lot-by-lot basis to assure that the normal variations occurring in these commercial materials do not reduce the quality level of NERVA products. Appendix B includes the applicable AFF Procedure.

Master file documentation includes the Purchasing Department Specification requirements, Purchase Order requirements, supplier test reports, results of laboratory analyses, and test results from evaluation hardware. This documentation is reviewed by the cognizant Quality Engineer and the cognizant Process Engineer, and both concur jointly in the Receiving Inspection Release of incoming quantities of raw materials for use in the manufacture of deliverable hardware.

A concerted raw materials control program, operated within the WANL-TNR-088 interpretation of NASA Specification NPC 200-2, must be supported by qualified and controlled laboratories which require large expenditures of budgeted funds. Westinghouse has qualified two laboratories for the analysis of incoming raw materials: the Technical Services Laboratories of the Atomic Power Divisions at Waltz Mills and the Chemistry Laboratories of the Nuclear Fuel Division at Cheswick. No primary standards are available for materials

similar to NERVA raw materials from the National Bureau of Standards or other recognized standardizing agencies. Qualification is performed by initiating controlled interchange of samples among supplier laboratories and Westinghouse laboratories to obtain common agreement. Procedures and techniques are then fixed and applied to the control of incoming quantities of raw materials. Periodic interchange between Westinghouse laboratories provides assurance of laboratory reproducibility.

The fuel element matrix materials graphite flour, carbon black, and partially polymerized furfuryl alcohol resin are commercial materials in high volume production commanding extensive markets. The NERVA requirements for tightened characterization and reproducibility of these materials is beyond the scope of commercial, high volume markets. The progressive acceptance of NERVA raw material specifications by large volume producers has been continually increased by concerted, mutual action on the part of Corporate Purchasing Departments and NERVA Fuel and Materials. This acceptance of small volume, specialized materials by large volume commercial producers has been accomplished with only nominal increases in raw material costs.

The Westinghouse programs of maintaining inclusive bidders' lists, upgrading specifications, qualification of suppliers, and control of incoming materials have been responsive to continuing changes in program objectives. Throughout the period of rapid and marked upgrading of reactor performance requirements to meet NERVA objectives, Westinghouse has provided an adequate supply of qualified raw materials. Concurrent with major shifts in reactor performance objectives, there have been accompanying changes in emphasis on the relative importance of various raw material properties. A continuing, extensive effort has been supplied from NERVA Fuel and Materials to apply the full range of accumulated experience, personnel skills, and laboratory facilities to evaluation of raw material interactions with fuel element and support hardware performance. Purchasing Department Specifications have been promptly upgraded by Specification and Controls Activities as definitions were generated. Intensive surveys of potential bidders and negotiation of responsive bids to purchase requirements have been continuously provided by the Purchasing Department. During any given period, raw material specifications have been satisfactory within the defined requirements at that time.

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The continued upgrading of reactor performance objectives now demands fuel element performance beyond any previous criteria. Raw materials specifications will continue to be upgraded and tightened. This demand will be fully supported by the inclusive materials evaluation, Purchasing Department market survey and order negotiation, supplier qualification, and incoming materials controls programs applied to NERVA raw materials.

2.0 MATRIX MATERIALS

2.1 GRAPHITE FLOUR

Original Westinghouse specifications for graphite flour were derived from characterizing the properties of material supplied by LASL at the onset of the NERVA Program. The intent of these specifications through PDS-30046, Revision B, was to provide a product within a selected range of the normal commercial control bands of volume production, having material properties suitable for application to NERVA fuel elements. Qualified suppliers were the Great Lakes Carbon Company and the Speer Carbon Company. From the outset, exact product repeatability was not demonstrated among sequentially purchased quantities. This was evidenced by processing variables in producing fuel elements.

By CY 1966, retention samples were available from a number of previously purchased quantities of graphite flour with well-characterized manufacturing histories. These quantities evidenced the complete range of processing variables encountered in producing fuel elements. During CY 1966, Westinghouse initiated a program to make a definitive characterization of the material properties of seven commercial quantities in terms allowing direct correlation to processibility. The successful conclusion of this program resulted in the issuance of PDS-30046, Revision C.

Previous revisions of PDS-30046 specified the normal properties characterized in commercial production of graphite flours: secondary graphite obtained from selected petroleum coke starting materials, density of billets used as grinding stock, maximum moisture content, particle size distribution of the ground flour, and maximum allowable impurities. PDS-30046, Revision C, retains these normal commercial requirements and, in addition, requires control within defined limits of product surface area, crystallographic interlayer spacing, and isotropy and micrograph structure as evidenced by micro-photographic techniques. The specification also places an in-process requirement on the supplier to control the maximum cross-sectional area of the extruded billets and the minimum graphitization temperature.

Westinghouse initiated an invited conference during January 1967 which was attended by representatives of eight commercial graphite producers: Asbury Graphite Mills, Stackpole Carbon, Pure Carbon, Speer Carbon, Great Lakes Carbon, Union Carbide Corporation,

Ultra Carbon, and Carborundum. The conference was held to convey the requirement for a small volume, special material for NERVA application. Potential suppliers indicated that commercial sources would supply closely specified flour.

Westinghouse then surveyed the graphite industry by invitations to quote against Purchasing Department procurement actions to the requirements of PDS-30046, Revision C. Extended negotiations were required, during which it was necessary for Westinghouse to provide complete data demonstrating that commercially produced material met the requirements of PDS-30046, Revision C, to establish a supplier willing to accept the product requirements imposed beyond normal commercial practice as a part of a Purchase Contract. This intensive effort was successfully concluded by placing a Purchase Order with the single responsive bidder, Great Lakes Carbon Company. Periodic surveying of all potential commercial sources for graphite flour will be continued by the Purchasing Department in an attempt to develop additional commercial sources of material to the requirements of PDS-30046, Revision C.

The Westinghouse program leading to the definitive characterization of commercial graphite flours previously applied to NERVA fuel elements was one of extended scope. This effort has been continued past the previously applied materials. This program includes consideration of alternative sources for starting cokes and processing technology to produce candidate graphite flours for evaluation in NERVA fuel elements. It includes a number of higher expansion graphites, very fine grained starting material for secondary graphite, and custom blending of particle size distribution.

2.2 CARBON BLACK

Westinghouse specification PDS-30047, Revision B, for carbon black is oriented toward commercial material in volume production in the carbon black industry. The material applied to NERVA fuel elements is the trade-named product THERMAX and is obtained from the qualified supplier, the R. T. Vanderbilt Company. Different grades, Regular THERMAX, P-33, and Stainless THERMAX, have been evaluated for product application. The regular grade continues to be specified because of lower permeability and lack of internal flaws in manufactured fuel elements.

Regular THERMAX is produced in large volume to a relatively wide range of process control parameters fully adequate for normal commercial applications. Westinghouse and the supplier have mutually explored a means of establishing better assurance of product uniformity in the material delivered for application to NERVA fuel elements. An agreement has been negotiated that the supplier will not ship Westinghouse material from warehouse stock. Westinghouse applies the Quality Surveillance requirement, which is a part of all Purchasing Department raw material specifications, to orders for carbon black. The supplier packages Westinghouse material from current plant output under surveillance. The supplier allows the Westinghouse representative into his proprietary plant control area on an informational basis during production of the material. The assurance that the supplier verifies his in-process control for each purchased quantity leads to increased product uniformity among the lots of material delivered against Westinghouse purchase orders. Acceptance criteria, in addition to supplier verification of process parameters, are based on maximum particle size, maximum allowable impurities, particle density, maximum moisture content, and maximum number of high density inclusions detectable radiographically.

2.3 PARTIALLY POLYMERIZED FURFURYL ALCOHOL RESIN

Westinghouse purchases the partially polymerized furfuryl alcohol resin (trade-named VARCUM) from the qualified supplier, Varcum Chemical, to the requirements of PDS-30048, Revision B. A program to make a definitive characterization of the resin, establishing controlled product properties and achieving uniform trouble-free performance during processing of fuel elements, was initiated and completed by Westinghouse during CY 1963. The results of this program, reported in WANL-TNR-132, are incorporated into PDS-30048. Acceptance criteria are based on solids content, viscosity, gel time, pH, and boron content. Quantities delivered by the supplier against PDS-30048 have been uniform in product properties.

Westinghouse will continue to conduct surveys and preliminary evaluations of modifications of furfuryl alcohol resins which polymerize by condensation-type reactions. Resin systems which polymerize by the addition-type reaction will be surveyed as potential candidate materials. This type of system eliminates the volatile reaction products, such as water, which result from the condensation-type polymerization reactions.

2.4 MALEIC ANHYDRIDE

Purchase of maleic anhydride to the requirements of PDS-51300-AB has been conducted within the policy of close cooperative interchange of information with the supplier. Assurance of product quality has been increased by negotiating the requirement with Fisher Scientific Company that the quantities shipped on Westinghouse purchase orders are obtained directly from the Fisher Fairlawn Plant and not from warehouse stock. A letter of conformance is on file from their Chief Chemist certifying that in-process samples, representative of the material, meet or exceed the requirements of chemical purity for recognized standards such as the U. S. Pharmacopoeia.

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3.0 FUEL BEADS

3.1 PYROCARBON-COATED URANIUM DICARBIDE FUEL BEADS

Westinghouse specifications for pyrocarbon-coated uranium dicarbide fuel beads were oriented toward control of the supplier's manufacturing processes through revisions up to and including PDS-30050, Revision A. These process-control-oriented specifications were continually updated to the latest fabrication information from all sources, including the various SNPO-sponsored Coated Particle Conferences.

The issuance of PDS-30050, Revision B, in CY 1965 introduced the concept to Westinghouse suppliers of control of pyrocarbon-coated uranium dicarbide particles wholly on a product specification basis. Revision B marked the first requirement of a 2300°C, 4-hour thermal migration test as a prime evaluation of fuel bead quality. This thermal migration requirement was developed and specified through close cooperative interchange of product properties and NERVA fuel element requirements between Minnesota Mining and Manufacturing and Westinghouse. Acceptance criteria, in addition to the thermal migration requirement, are based on weight percent uranium versus coated particle density, screen size distribution, acid leachable uranium, particle shape, and maximum allowable impurities.

Shortly following the qualification of Minnesota Mining and Manufacturing according to the product requirements of PD-30050, Revision B, sufficient information and product samples were interchanged between Westinghouse and two other suppliers, the Carbon Products Division of the Union Carbide Corporation and Gulf-General Atomic (formerly General Atomic Division, General Dynamics Corporation), to qualify both according to PDS-30050, Revision B. Commercial quantities have been received, tested, and certified according to PDS-30050, Revision B, requirements from all three suppliers.

Westinghouse has maintained a detailed follow of the properties of each incoming lot of fuel beads with continuous feedback, where required, to fuel bead suppliers, leading to improved control of supplier testing, control of minor rare earth impurities, and product repeatability within narrow control bands. PDS-30050 has undergone Revisions C, D, and E, based on the inputs from continuing cooperative information exchange between Westinghouse and product suppliers. This interchange has resulted in a progressive increase in product quality.

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The Purchasing Department is presently re-surveying potential commercial sources for pyrocarbon-coated fuel beads by issuing invitations for quotation against the current product specification, PDS-30050-2, Revision E. This is part of the routine program of maintaining inclusive, up-to-date bidder lists. Minnesota Mining and Manufacturing and the Carbon Products Division of The Union Carbide Corporation both presently indicate they are not to be considered active supplier candidates. Nuclear Materials and Equipment Corporation indicates interest in becoming a potential supplier at a future date but not at present. Nuclear Fuel and Services has not responded to the invitation for quotation at this time. Gulf-General Atomic is presently qualified and shipping product to the requirements of PDS-30050-2, Revision E.

Westinghouse has maintained a detailed, current knowledge of available information concerning pyrocarbon-coated uranium dicarbide fuel beads from all sources. The fuel bead development work at the Los Alamos Scientific Laboratory, summarized at the SNPO-sponsored Thirteenth Coated Particle Conference, demonstrated that further major pyrocarbon-coated product improvements can be obtained on a laboratory scale. The two major improvements are increase of thermal migration resistance from the 2300°C region to the 2600°C region and the formation of an internal void between the uranium dicarbide core and the pyrocarbon shell. The internal void is of sufficient magnitude to minimize the effect of the significantly higher coefficient of thermal expansion of the uranium-containing fuel bead core. Unless absorbed, this expansion would cause serious tensile stresses in the fuel bead pyrocarbon coating at the reactor operating temperatures projected for future NERVA requirements.

Westinghouse recently proposed and obtained approval of a program to enter into negotiation with the current fuel bead supplier, Gulf-General Atomic, to explore the feasibility of transferring the LASL Laboratory results to a commercial scale and obtaining a sufficient quantity of material to manufacture qualification fuel elements. Preliminary results reported from the Gulf-General Atomic Laboratories indicate that both the 2600°C thermal migration resistance and the internal void for stress relief can be achieved using commercial equipment.

The effort of maintaining close contact with all external sources of information concerned with any variations of the uranium-carbon system will continue. The extensive internal effort to define the properties and correlate the performance of products delivered by suppliers will also be continued. Programs will be initiated, as required, to demonstrate upgraded fuel bead quality to the requirements of the NERVA program.

4.0 FUEL ELEMENT TIP MATERIALS

4.1 MOLYBDENUM BRAZING POWDER

The molybdenum powder for tip brazing is purchased to the requirements of PDS-30179, Revision A, from the qualified vendor, Fansteel Metallurgical. Material obtained from Sylvania has been evaluated by NERVA Fuel and Materials and found equivalent during development efforts. Sylvania is an alternative source for this material in the event one should be required. Acceptance criteria are based on maximum particle size, minimum assay, and maximum allowable impurities.

4.2 CARBIDE/GRAPHITE COMPOSITE MATERIAL

The carbide/graphite composite material containing 75 w/o NbC is purchased to the requirements of PDS-30106, Revision E, from the qualified suppliers, the Carborundum Company and the Norton Company. The material is fabricated from the niobium carbide powder specified in PDS-30104, Revision E, and the carbon powder specified in PDS-30106, Revision E. Acceptance criteria are based on homogenity, density, flexural strength, elevated temperature creep deformation, and thermal expansion characteristics. Material purchased from the Donald W. Douglas Laboratories, Supertemp Corporation, and Union Carbide Corporation, and Union Carbide Corporation have not yet met qualification requirements.

4.3 CARBON POWDER

Carbon powder supplied by the Union Carbide Corporation and the Carborundum Company, as specified in PDS-30106, Revision E, has been utilized in the development and qualification of carbide/graphite composite material. Acceptance criteria are based on the selection of "needle" coke with elongated particle shape, maximum limit of impurities, and particle size.

Efforts to define carbon powder requirements more accurately, with respect to carbide/graphite composite material properties, will be continued. This effort will establish whether specification upgrading is necessary by further definition of raw materials properties.

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4.4 NIOBIUM CARBIDE POWDER

Niobium carbide powder is purchased to the requirements of PDS-30104, Revision E, from the qualified supplier, Kawecki Chemical Company. Material obtained from the Wah Chang Company, Fansteel Metallurgical Company, and Kenametal has not yet met supplier qualification requirements. Acceptance criteria are based on minimum assay, apparent density, tap density, maximum limit of impurities, particle size, and porosity.

The effects of niobium carbide powder variations on the properties of carbide/graphite composite material are presently being evaluated. This effort will be continued to establish whether PDS-30104, Revision E, will require upgrading by reducing allowable impurities limits.

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5.0 COATING MATERIALS

5.1 NIOBIUM (COLUMBIUM) PENTACHLORIDE

Two commercial sources of niobium pentachloride, one domestic (Stauffer Chemical Company) and one foreign (Atomergic Chemical), have been qualified by Westinghouse to the requirements of PDS-30052, Revision D. Acceptance criteria are based on minimum assay, maximum nodule size, maximum allowable impurities, and pressure above atmospheric of the protective atmosphere maintained in the shipping containers.

The continuous surveillance of the market place, a joint Purchasing-NERVA Fuel and Materials effort, allows Westinghouse to determine any changes in potential sources of supply. As a result, the entry of the Wah Chang Company into the field of quantity suppliers was noted. A small quantity of the commercial production from the Wah Chang Company is being purchased to attempt to qualify another domestic supplier to specification requirements.

Westinghouse has applied intensive continued cooperative interchange of raw material information with the qualified suppliers, both by negotiations of the Purchasing Department and implementing the Quality Surveillance requirement of PDS-30052, a part of all Westinghouse raw material specifications, in a supplier's plant. This effort has resulted in satisfactory packaging of the product, leading to assurance of lack of product degradation during extended storage, a progressive reduction in oxygen contamination of the product, and control of the maximum mesh size of nodules shipped by the suppliers.

5.2 ZIRCONIUM TETRACHLORIDE

A Purchasing Department Specification, PDS-30131, requiring material of minimum purity equal to the commercially available re-sublimed grade, has been prepared. The experience gained in developing packaging requirements for niobium pentachloride is being drawn on for this specification. Acceptance criteria are based on minimum assay, maximum particle size, maximum allowable impurities, and pressure above atmospheric of the protective atmosphere maintained in the shipping containers.

Supplier qualification will be performed during the PeWee-2 pre-production period.

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5.3 MOLYBDENUM HEXACARBONYL

The use of molybdenum overcoating in the bores of NERVA fuel elements and any resulting association with the compound molybdenum hexacarbonyl is defined by the NERVA Classification Guide as Confidential-Restricted. To prevent disclosure of the association of molybdenum hexacarbonyl with the NERVA program, Westinghouse purchases the material as the commercial grade with the requirement that total impurities are not to exceed 1000 ppm. Climax Molybdenum was previously qualified as a supplier.

Due to processing equipment limitations, the supplier has delivered the material in small (5 to 10 pounds) manufacturing lots. Westinghouse cross-blends these small manufacturing lots into 100-pound or larger master lots in a double-cone blender reserved for use with molybdenum hexacarbonyl. Thus, the blender is eliminated as a potential source of contamination of the material. Samples representative of the entire blender content, taken at the time of re-packaging, are analyzed and the material upgraded to the classified specification PDS-30095. Acceptance criteria are based on maximum allowable impurities, minimum assay, minimum insoluble residue in chloroform, and X-ray diffraction patterns typical of molybdenum hexacarbonyl.

The large master lots of homogeneous, well-characterized material provide the ability for both process qualification and manufacture of all fuel elements for a given core from a stable, well-known material source.

Climax Molybdenum has recently encountered difficulty with processing equipment used for the manufacture of molybdenum hexacarbonyl. This equipment operates at high pressures and an unsafe condition has developed. The company cannot manufacture molybdenum hexacarbonyl at present. Numerous contacts with potential suppliers by Westinghouse could jeopardize the classified NERVA association with molybdenum hexacarbonyl. To alleviate the risk of this disclosure, an agreement was negotiated with Climax Molybdenum to develop another source for the material by conducting a market survey and acting as a procurement agency for Westinghouse. Evaluation of four potential suppliers, one foreign (Atomergic)

and three domestic (Wah Chang, Var-Lac-Oid, and Pressure Chemicals), by Climax Molybdenum technical personnel conversant with the production of molybdenum hexacarbonyl led to the conclusion that the material available from Pressure Chemicals is similar to that previously supplied Westinghouse from Climax Molybdenum facilities. This conclusion is based both on manufacturing processes and material properties.

Climax Molybdenum is presently supplying Westinghouse with molybdenum hexacarbonyl manufactured by Pressure Chemicals. This material will be qualified during the PeWee-2 pre-production period.

5.4 ANHYDROUS HYDROGEN CHLORIDE

Recurring problems with surface finish and structure of niobium carbide coatings during CY 1964 were established as being associated with high (to 1200 ppm) and variable content of phosgene and acetylene in the anhydrous chloride delivered by the Matheson Company. Close cooperative interchange of information with the supplier established that the hydrogen chloride was a by-product of a vinyl chloride manufacturing operation and that high and variable organic content is inherent in such material. An extended survey of some twenty potential suppliers conducted by the Purchasing Department established that most commercial hydrogen chloride is a by-product of the vinyl chloride process and it is not commercially feasible to remove the organic contaminants. It was further established that chemically pure, reagent grade hydrogen chloride was in short supply and very expensive. One responsive bid was obtained from Frontier Chemical for anhydrous hydrogen chloride produced by an inorganic reaction. PDS-52217-AP (Sub 3) was issued and the hydrogen chloride from Frontier Chemical qualified in Westinghouse processes. Early deliveries from this supplier established that occasional lots suffered from high moisture content. Intensive supplier conferences caused Frontier Chemical to introduce in-process controls assuring the delivery of dry (less than 100 ppm water), organic-free anhydrous hydrogen chloride. Acceptance criteria are based on minimum assay and maximum allowable impurities.

The recent introduction of an electronic grade anhydrous hydrogen chloride by Air Products on a commercial scale was followed by procurement of a sufficient quantity for qualification in Westinghouse processes. The qualification was successfully completed but it was not possible, at the time, to establish any significant advantage in using the more expensive (a cost increase by more than six times) electronic grade anhydrous hydrogen chloride rather than the commercial grade being procured from Frontier Chemical. Air Products is a qualified supplier of the electronic grade anhydrous hydrogen chloride. This allows a rapid and direct process application at any time the higher purity gas is needed for the fuel elements of the future.

5.5 INERT CARRIER GAS

Westinghouse originally applied helium as the inert carrier gas in fuel element manufacturing processes. Continued difficulty was encountered with contaminants, primarily oxygen and moisture from leaky U.S. Navy trailers and hydrocarbons from the "oil pumped" equipment used in compressing large quantities of helium. The variability in delivered product, the fact that helium is a gas in relatively critical supply, and budget considerations led Westinghouse to evaluate the substitution of argon in place of helium as the inert carrier gas.

The successful evaluation and qualification of argon in fuel element manufacturing processes was followed by the installation of a liquid argon supply system. The attendant lower cost and higher product purity have provided consistently high purity inert carrier gas from all purchased quantities delivered by the qualified supplier, Air Products, according to the requirements of PD S-52118-Ba (Sub A). Acceptance criteria are based on minimum assay and maximum allowable impurities.

5.6 HYDROGEN

Westinghouse has procured electronic grade hydrogen from the qualified vendor, Air Products, in accordance with the requirements of PDS-3057-F-3. Quantities delivered by the vendor against the specification have been uniform in product properties. Acceptance criteria are based on minimum assay and maximum allowable impurities.

5.7 METHANE

Westinghouse has purchased methane from the qualified vendor, Air Products, as the chemically pure grade. A joint Purchasing-NERVA Fuel and Materials survey of the purity of commercially available material has been completed. A Purchasing Department Specification PDS-30130 is being prepared specifying material with a minimum purity equal to the commercially available ultra-pure grade. Supplier qualification will be performed during the PeWee-2 pre-production period. Acceptance criteria are based on minimum assay and maximum allowable impurities.

6.0 FUEL ELEMENT KEY MATERIAL

6.1 KEY BRAZING POWDER

The alloy powder used in brazing keys to fuel elements is purchased in accordance with the requirements of PDS-30177 from the qualified supplier Brush Beryllium. Brush Beryllium is the only known commercial source for this brazing alloy. Acceptance criteria are based on particle size, composition, and maximum allowable impurities.

APPENDIX A

Quality Methods and Procedures (QMP's) Defining the Receiving Inspection and Release of Raw Materials

QUALITY METHODS & PROCEDURES WESTINGHOUSE ASTRONUCLEAR LABORATORY Westinghouse Electric Corporation P.O. Box 10864, Pittsburgh, Pa. 15236	QMP No. 4 - 3 Rev. ____ Effective Date July 25, 1966 Supersedes: No. QMP(L)4-3 Date 12-15-65

SUBJECT: IDENTIFICATION OF MATERIAL, DETAIL PARTS, SUBASSEMBLIES, AND ASSEMBLIES BY SERIAL OR LOT SERIAL NUMBERS

1.0 PURPOSE

To establish a procedure for assigning non-repeating individual or lot serial numbers to material, detail parts, subassemblies, and assemblies for complete traceability and control.

2.0 APPLICABILITY

The following procedure shall be applicable to individual or lot serial number identification of all material, detail parts, subassemblies manufactured or purchased by WANL, WAFF, or applicable subcontractors. Government-Furnished Property (GFP) is an exception since it is identified in accordance with Government regulations.

3.0 REFERENCE

QMP No. 5-3, Quality Surveillance Codes

4.0 GENERAL

4.1 Policy for WANL Serial Number Implementation

Material Control at WANL is responsible for establishing and assigning serial numbers on purchase requisitions, IWR's, and work orders. The requirement for identifying parts, subassemblies, and assemblies by individual or lot serial numbers shall be indicated on the drawing. When a serial number is required, the drawing shall also specify the place on the part where the serial number shall be applied, and the manner in which it shall be applied. Expendable-type standard hardware, such as nuts, bolts, washers, rivets, fasteners, etc., not defined by WANL drawings do not require WANL serialization unless specified on the ordering document. These parts may be defined by Military drawing or specifications (such as AN or MS drawings) or by standard catalog numbers.

APPROVALS:

WANL Q. C. Mgr.	WAFF Q. C. Mgr.	Product Assurance Mgr.	Program Manager

4.2 Policy for WAFF Serial Number Implementation

WAFF Inspection is responsible for establishing and assigning serial and lot numbers for all raw materials and components requiring control. The responsibility for establishing and assigning particular serial of serial and or lot numbers may be delegated by Inspection to other departments with their agreement. The ultimate responsibility for the adequacy of such delegated responsibility rests with Inspection.

4.3 Definitions

4.3.1 Lot

A group of materials or parts of the same design (Part Number, Drawing Revision, Specification, etc.) consisting of homogeneous groups of materials or parts, produced under essentially the same conditions of manufacture and time.

4.3.2 Lot Serial Number

A unique number assigned to a lot for the purpose of identification.

4.3.3 Serial Number

A unique number assigned to a distinguishable part for the purpose of identification.

5.0 PROCEDURE FOR SERIALIZATION AT WANL

5.1 Assignment of Serial Numbers

Individual or lot serial numbers, when so indicated by the drawing requirements, shall be pre-assigned in non-repeating consecutive sequence by Material Control on the purchase requisitions, IWR's and work orders. Material Control shall originate and maintain a log showing serial and lot serial numbers assigned against purchase requisition, work order, or IWR numbers and part numbers. When appropriate and approved by Material Control, suppliers' serial numbers may be used in lieu of WANL-assigned numbers.

5.1.1 Addition of Parts to Orders

When serial-numbered parts are added to an existing order, Material Control shall assign new serial numbers, as required. These numbers shall be placed on the Request for Change Notice, which shall be transcribed on the Change Notice by Purchasing. When lot serial-numbered parts are added to an existing order, new lot serial numbers shall be assigned to the additional parts. All additions of parts to orders shall be noted by Material Control in the logbook.

5.1.2 Deletion of Parts from Orders

When serial-numbered parts are to be deleted from an existing order, Material Control shall note on the Request for Change Notice the quantity of parts that are to be cancelled and shall request that Purchasing advise, on the Change Notice, the particular serial numbers that are to be voided. The buyer in the Purchasing Department shall coordinate with the WANL supplier to determine which serial numbers shall be voided. When lot serial-numbered parts are to be voided from an existing order, Material Control shall note on the Request for Change Notice the quantity of parts that are to be voided and the lot serial numbers involved. All voided serial numbers for cancelled parts on order shall be noted by Material Control in the logbook and are not to be reassigned.

5.1.3 Repair or Replacement of Parts Returned to Supplier

When parts are returned to the supplier for repair or replacement, the serial number will remain unchanged if the parts are repaired and subsequently returned to WANL. If the supplier chooses to scrap the rejected parts and replace with new parts, the WANL buyer must be contacted in order to provide a new serial number. The WANL buyer shall obtain the serial numbers from Material Control. In no instance will a rejected part serial number be assigned to a replacement part.

5.2 Multiple Part Raw Material Identification

5.2.1 WANL-purchased raw material which will be in the form of bars, sheet, plate stock, and castings or forgings which will be processed into multiple detail parts shall be identified by the manufacturer's heat number, furnace run, batch number, melt number, etc. (WANL serial numbers are not required for raw material procurement except when specified by drawing.)

5.2.2 Supplier-furnished raw material to be processed into multiple parts shall be traceable to the manufacturer's heat number, furnace run, batch number, melt number, etc. Shipping documentation of detail parts manufactured from this raw material will carry reference to the raw material identification.

5.3 Single Part Raw Material Identification

Individual castings, forgings, etc., which will be manufactured or processed, such that each will make a single detail part, will be assigned a WANL serial number at the raw material source as described in paragraph 5.4.1, below. This number will remain with the part through all subsequent operations, regardless of where performed. If the number is removed by machining or processing, it will be replaced as soon as possible by the supplier performing the operation.

5.4 Method of Serial or Lot Identification

5.4.1 Individual Part Serial Numbers Numbering System

Serial numbers shall consist of a letter "A" prefix followed by consecutive numbers, starting with "1". This method shall be followed until the numbers reach "99999," after which the letter prefix shall be changed to "B", again starting with the number "1". This shall be continued through the alphabet and thereafter, if required, "AA, AB, AC" etc., and "BA, BB, BC" etc. shall be used as a prefix. The letters "I", "O", "Q", "L", and "X" shall not be used.

Examples:

P/R 30314 130 pieces	P/N 909E421HO1 S/N A21 through A150
P/R 41679 200 pieces	P/N 945C967HO1A S/N A151 through A350

5.4.2 Lot Serial Numbers

5.4.2.1 Numbering System

Lot serial numbers shall be assigned in the same manner and series as individual serial numbers, except that the prefix letter "L" shall be a part of each lot serial number.

Examples:

P/R 30642 10,000 pieces (10 lots)	P/N 936J654HO1B Lot S/N LA1 through LA10
P/R 41647 5,000 pieces (5 lots)	P/N 945C852HO2C Lot S/N LA11 through LA15

5.4.2.2 Assignment of Numbers

Assignment of lot serial numbers shall not violate the rule that the parts within a lot shall not be mixed with respect to raw material, batch, heat, furnace, or melt number. The assignment of a series of lot serial numbers will be coordinated with the supplier by WANL Purchasing in order to determine an objective lot size consistent with raw material lots, manufacturing process, shipping schedule, and order size.

5.5 Effect of Changes Involving Existing Parts that have been Previously Serialized

5.5.1 Individual and Lot Serial Numbers (not involving multiple part changes)

Drawing or part number changes which require modification and/or re-identification shall have no effect on individual or lot serial numbers. The original serial number assigned shall be retained and shall not be obliterated or altered.

5.5.2 Individual Serial Numbers (changes involving multiple part numbers)

One individual serialized part reworked or modified and re-identified into two or more new part numbers shall have a new serial number assigned for each piece. The new serial numbers are to be assigned and recorded in the logbook by Material Control.

5.5.3 Lot Serial Numbers (changes involving multiple part numbers)

All parts previously lot serialized which are subsequently divided, segregated, reworked, modified, and/or re-identified into two or more new parts shall retain the original lot serial number.

6.0 PROCEDURE FOR SERALIZATION AT WAFF

6.1 Assignment of Serial or Lot Numbers at Receiving Inspection

Lot or serial numbers shall be assigned by In-Process Inspection to all raw materials and components received at Receiving Inspection, with the exception of materials or parts in Quality Surveillance Class 3 or 4 (see QMP No. 5-3). The lot and serial numbers shall consist of a two-letter supplier code, assigned by In-Process Inspection, followed by consecutive numbers starting with "1", to a maximum of four digits. A master log shall be established by In-Process Inspection, consisting of a log sheet(s) for each supplier. Serial and lot numbers shall be assigned consecutively, as required, for all materials and parts received, continuing from P.O. to P.O. Each log sheet shall have, as a minimum, the following headings:

WAFF Lot/Serial Numbers
WAFF P. O. Number
WAFF Design Requirement (Part Number, Drawing/Revision, Specification, etc.)
Part Name
Quantity (Applicable to Lot Numbers only)
Supplier Name
Supplier Lot Number
Supplier Certification Number
Container Identification Numbers (see paragraph 6.1.1, below)

6.1.1 Container Identification Numbers

In addition to lot numbers, container numbers shall be assigned to all lots of material requiring individual container control through pre-processing. The container numbers shall be consecutive numbers, starting with "1" for each lot of material. If the supplier has assigned container identification numbers, those numbers shall be utilized, rather than assigning new numbers.

6.2 Serial and Lot Numbers Assigned During Manufacture at WAFF

Because of its classified and changeable nature, the information required for this paragraph will not be distributed with the Quality Control Manual. Concerned individuals may refer to WAFF Quality Engineering to obtain this information.

6.3 Recording of Assigned Serial and Lot Numbers

In addition to the master log required by paragraph 6.1, above, each Quality Control form, tag, etc., shall have provisions for recording serial and lot numbers, as required, to assure identification and traceability.

7.0 IMPLEMENTATION BY WANL AND WAFF SUPPLIERS

WANL Quality Control Representatives during visits to suppliers shall assure that subcontractors correctly mark raw material, detail parts, subassemblies, and assemblies with the required raw material, serial, or lot serial numbers. The serial numbers shall be marked on the parts at the earliest possible point of manufacture, and these numbers shall be maintained. If serial numbers are removed by manufacturing operations, they shall immediately be replaced as originally required. All documentation originated by WANL suppliers shall reference the actual serial number or lot serial number involved. The terminology "all affected" shall never be used. If the documentation is intended to apply to the entire order, then the numbers will be listed in the following manner: "serial number A150 through A950". Shipping documents which accompany each shipment of parts shall reference the raw material identification numbers and shall correlate such numbers with the actual serial or lot serial numbers manufactured from each lot of raw material.

8.0 SYSTEM FOR ASSURING NON-DUPLICATION OF SERIAL AND LOT SERIAL NUMBERS AT WANL

The Serial and Lot Serial Numbers assigned by Material Control are reviewed by Quality Quality Control to assure that duplication does not exist. Upon receipt of the material and parts at WANL, Quality Control will verify that no serial or lot serial numbers have been used by the supplier which were not assigned and that there is no duplication of numbers.

QUALITY METHODS & PROCEDURES	QMP No. 5-3 Rev.
WESTINGHOUSE ASTRONUCLEAR LABORATORY Westinghouse Electric Corporation P.O. Box 10864, Pittsburgh, Pa. 15236	Effective Date July 25, 1966
	Supersedes: No. QMP(AL)5-3 Date 12-15-65

SUBJECT: QUALITY SURVEILLANCE CODES

1.0 PURPOSE

To define the procurement Quality Surveillance Codes, establish related responsibilities and identify the degree of surveillance to be exercised by Quality Control over various phases of design, procurement, testing, etc. as may be required.

2.0 REFERENCE

QMP No. 4-1, Drawing Review
QMP No. 5-2, Quality Requirements for Procurement Documents
QMP No. 8-1, Nonconforming Material Disposition
QMP No. 8-2, Variation Requests
Divisional Procedure No. 300GO, "Engineering Drawing Codes"

3.0 GENERAL

The quality surveillance exercised on a given item, from the initial design stage through delivery and end use depends largely upon the status of the design and the quality confidence level desired. The degree of surveillance required establishes the procedures and controls necessary to monitor the various activities through design, procurement, receiving inspection, testing and shipping. This degree of Quality Control surveillance should be commensurate with the applicable contract quality plan objectives. This procedure shall apply to all purchase requisitions and orders, purchase requisition and order change notices, work orders and work order change notices as referenced in QMP No. 5-2.

4.0 PROCEDURE

4.1 Quality Surveillance Codes

As defined in the referenced WANL Divisional Procedure, all WANL drawings are prepared in accordance with the characteristics of a series of drawing codes, (Code 1 through Code 4) designed to depict drafting practices and drawing quality commensurate with the end use and/or contract requirements. These codes are shown in the title block on each drawing.

APPROVALS:

WANL Q.C. Manager	WAPD Q.C. Manager	Product Assurance Mgr.	Program Mgr.

The assignment of Quality Control Surveillance Codes shall be consistent with the Drawing Code designations. Under no circumstances will the assignment of Surveillance Codes be less than the designated drawing code. For example, Quality Surveillance Codes 1 or 2 may be specified for a Code 2 drawing, but Surveillance Codes 3 or 4 are not permitted.

Where items are not covered by WANL drawings, the degree of Quality Control coverage shall be determined by evaluating the end use requirements, the interface objectives, the quality level desired and selected in accordance with the following Quality Surveillance Code descriptions:

4.1.1 Surveillance - Code 1 and Code 2

Includes all items designated and ordered for engineering and contractual development tests and all related support equipment which could affect the quality of the test effort, the test system reliability and the results of the test program. It also includes all items described by drawings and/or specifications as well as all articles to be delivered to customer and/or test sites where reliability and quality are to be maintained at the highest levels.

Typical Examples -

Drawing Code 1	Drawing Code 2
End Item Control Systems	Test Car Assembly
Shipping Containers	Electronic Checkout Systems
Interface Control Assemblies	Instrumentation (diagnostic data)
Test Articles (top assembly)	Critical Assembly and Disassembly tools, fixtures and non-reactor/test articles.
Test Article Backup and Raw Material Items	Test fixtures and test hardware having safety and reliability consideration
Liquid Metal Pumps	Lifting and handling fixtures
Space Radiators	Test Facility Pressure Vessels
Reactor Hardware (including backup and experimental parts)	Reactor Pressure Vessels
High Pressure Piping	Ground Support Nuclear Instrumentation and Radiation Safety Devices
High Temperature Facilities	Customer End Items
Test Car and Piping Assembly	
Turbo-generator Hardware	
Instrumentation for Reactor and other Test Article Diagnostic Data	

Degree of Surveillance - The type and degree of Quality Control Coverage shall be the same for both Surveillance Codes 1 and 2. Items falling under these codes shall receive full Quality Control coverage as determined by the cognizant Quality Engineer.

All drawings designated as Codes 1 and 2 shall be reviewed and signed by Quality Control in accordance with referenced procedure QMP No. 4-1.

Deviation and waiver requests shall be submitted for disposition in accordance with referenced procedure QMP No. 8-2.

Nonconforming parts will be submitted to the Engineering Review Board for disposition in accordance with referenced procedure, QMP No. 8-1.

Nonconformities on End Item assemblies will be submitted to full Material Review Board for disposition.

4.1.2 Surveillance Code 3

Includes all hardware, raw materials and assemblies designated for development and experimental engineering tests and evaluation. Also includes all related instrumentation, support equipment, hardware and non-critical assembly and disassembly tools intended for use at WANL which are procured to commercial specifications, where the reliability and quality of such items can have a direct affect on the evaluation results.

Typical Examples -

Capital equipment such as Instrumentation and measuring devices.	Facility tools and safety monitoring devices.
Gauges (non-interfacing)	Mock ups
Non-critical Assembly and Disassembly tools.	ASME Coded Pressure Vessels
Component Containers	Impregnated graphite
	Neutron moderators

Degree of Surveillance - The requisitioner will be responsible for defining in the ordering documents the desired Quality Control coverage. The cognizant Quality Engineer will review all Code 3 procurements to ensure that the degree of quality control specified can be provided and is consistent with the objectives of the test program. Quality Control procedures and documents will be initiated where necessary.

Drawings designated as Code 3 will be reviewed and signed by Quality Control when requested by the cognizant design engineer in accordance with referenced procedure QMP No. 4-1.

Where Quality Control procedures and documents are implemented, deviations, waivers and nonconforming parts shall be dispositioned in accordance with referenced procedures, QMP No. 8-1 and QMP No. 8-2.

4.1.3 Surveillance Code 4

Limited to standard items or supplies for facility maintenance and services where the quality of such items will not affect the performance or reliability of the products being supplied to WANL customers. The requisitioner will be responsible for making this determination.

Degree of Surveillance – Quality Control input will not be provided Code 4 drawings will not be reviewed or signed by Quality Control.

QUALITY METHODS & PROCEDURES WESTINGHOUSE ASTRONUCLEAR LABORATORY Westinghouse Electric Corporation P.O.Box 10864, Pittsburgh, Pa. 15236	QMP No. 5 - 4 Rev. ____ Effective Date 7/25/66 Supersedes No. QMP(L) 5-4 Date 10/1/65 QMP(A)T4-3 10/1/65

SUBJECT:

CORRECTIVE ACTION AT SUPFLIERS

1.0 PURPOSE

To establish a procedure for obtaining corrective action for all external discrepancies and deficiencies.

2.0 APPLICABILITY

This procedure shall be followed whenever, in the estimation of the cognizant Quality Control organization, corrective action for external discrepancies and deficiencies is necessary.

3.0 REFERENCE

QMP 8-2, Variation Request

4.0 GENERAL

4.1 Policy

It is the responsibility of Supplier Surveillance at WANL and Quality Engineering at WAFF to take prompt action to correct conditions at suppliers' plants which have resulted or might result in substandard or defective materials, parts, assemblies or services. Initiation of corrective action for fuel element suppliers is, however, an exception and shall be the responsibility of Product Control Engineering at WAFF. Disregard or unsatisfactory resolution of Request for Corrective Action (RCA) shall be cause for removal of a supplier from the approved procurement sources list.

5.0 PROCEDURE

5.1 The RCA shall be initiated whenever, in the estimation of the cognizant Quality Control organization, the following unsatisfactory conditions are found to exist and require formal corrective action.

APPROVALS:

WANL Q. C. Mgr.	WAFF Q. C. Mgr.	Product Assurance Mgr.	Program Manager

5.1.1 Supplier Quality Control written systems or procedures are inadequate to meet the requirements of WANL and WAFF drawings, specifications or other Quality Control requirements specified in the Purchase Order.

5.1.2 Supplier Manufacturing or Quality Control practices are detrimental to the quality of raw material or hardware produced for WANL and WAFF.

5.1.3 Supplier inspection results are found to be incomplete or incorrect as determined by Quality Control representatives.

5.1.4 Supplier hardware or raw material is found to be outside of drawing and specification limits (not previously authorized by Variation Request - see QMP 8-2) as determined by Westinghouse Receiving Inspection or subsequent use.

5.2 Preparation and Use of Request for Corrective Action (℈ Form No. 59072)

5.2.1 Originating the RCA

5.2.1.1 The WANL Supplier Surveillance representative or the Quality Engineer at WAFF will complete the top portion of the RCA as follows:

5.2.1.1.1 The date shall be the date on which the RCA is originated.

5.2.1.1.2 The RCA shall specifically describe the deficiency with regard to drawing numbers, items, characteristics, specification sections, serial numbers, as applicable.

5.2.1.1.3 Consequence of noncompliance shouid be briefly stated to supplier if not obvious.

5.2.1.2 The WANL Supplier Surveillance Supervisor at Large or the Quality Engineering Supervisor at WAFF shall maintain a log and assign a number to each RCA. The numbering system shall be consecutive, beginning with number one and progress numerically, regardless of purchase order number, for each supplier involved. In addition, he shall countersign the RCA in the space provided.

5.2.1.3 Four copies of the form will be sent to the cognizant buyer along with a cover letter signed by the initiating Engineer's Supervisor. The buyer shall forward three copies of the RCA to the involved supplier's Quality Control Manager for his reply.

5.2.2 **Supplier Responsibilities**

5.2.2.1 The supplier's Quality Control Manager shall complete the second part of the form. It should be noted that the expected completion date is required.

5.2.2.2 The supplier shall return two copies to the cognizant buyer who, in turn, will forward one copy of the RCA to the initiating Engineer's Supervisor.

5.3 **Corrective Action Follow Up**

5.3.1 Upon receipt of the RCA with the supplier's proposed corrective action from the cognizant buyer, the cognizant supervisor shall indicate his comments as to acceptance or rejection of the proposed corrective action. If the proposed corrective action is not acceptable, the RCA is returned to the cognizant buyer to be sent back to the supplier, stating that his corrective action has not been accepted by WANL and WAFF. In the event that an RCA is not acceptable, the procedures as outlined in Paragraphs 4.2.1.3, 4.2.2.1 and 4.2.2.2 will be followed.

5.3.2 Upon receipt of an acceptable proposed corrective action, the cognizant Quality Control Supervisor will notify the Westinghouse Quality Control representative who will follow through to verify suppliers compliance.

5.3.3 Unsatisfactory performance of the proposed corrective action shall be cause for the initiation of another RCA or eventual removal from the WANL and WAFF Evaluated Suppliers List.

QUALITY METHODS & PROCEDURES WESTINGHOUSE ASTRONUCLEAR LABORATORY Westinghouse Electric Corporation P.O. Box 10864, Pittsburgh, Pa. 15236	QMP No. 5 - 8 Rev. ____ Effective Date 7/25/66 Supersedes: No. QMP(A) 5-5 Date 10/1/65

SUBJECT: RECEIVING INSPECTION

1.0 PURPOSE

To establish the procedure and associated records to be utilized in Receiving Inspection.

2.0 APPLICABILITY

This procedure is applicable to all Receiving Inspection functions performed at WAFF.

3.0 REFERENCES

QMP No. 4-3, Identification and Traceability of Raw Materials and Components by Serial or Lot Numbers
QMP No. 5-3, Quality Surveillance Codes
QMP No. 7-2, Inspection and Test Planning
QMP No. 9-2, Design, Acquisition, Calibration and Control of Inspection Tools and Gages
QMP No. 5-4, Corrective Action at Suppliers

4.0 GENERAL

4.1 Policy

Quality Engineering is responsible for establishing the Receiving Inspections (including testing) required for all purchased materials in Quality Surveillance Class 2, or higher (see QMP No. 5-3), to assure that the purchase order requirements are met. In-Process Inspection is responsible for performing the Receiving Inspections as stated in the applicable Quality Inspection Procedures (QIP's). When discrepant materials are discovered at Receiving Inspection, they shall be moved, whenever possible, to an isolated area set aside for discrepant materials, under the control of Quality Control. No purchased material in Quality Surveillance Class 2, or higher, shall be utilized by Operations at WAFF prior to inspection and acceptance by Quality Engineering.

APPROVALS:

WANL Q. C. Mgr.	WAFF Q. C. Mgr.	Product Assurance Mgr.	Program Manager

4.2 Definitions

Receiving Inspection:

1) the process of determining acceptability or unacceptability of purchased materials, and

2) the control and dispositioning of raw materials through pre-processing operations, such as cross-blending and sifting of a raw material up to, but not including the formation of Dry Carbon Ingredients (DCI).

5.0 PROCEDURE

5.1 Receipt and Filing of Purchase Orders, Purchase Order Change Notices, and Supplier Test Reports and Certifications

A copy of each purchase order (and change notice) placed by WAFF shall be supplied to Quality Engineering by WAFF Purchasing. Upon receipt, Quality Engineering shall establish a file for each purchase order for the purpose of accumulating all documents applicable to that order. Purchasing shall also forward to Quality Engineering, upon receipt, a copy of each supplier test report and certification, which shall be retained in the applicable purchase order file.

5.2 Receipt and Inspection of Purchased Material (Except Fuel, Tools, and and Gages

5.2.1 Receipt of Purchased Raw Material

Upon receipt of purchased material (including samples), Receiving shall remove all excess packaging; determine that the materials are being received against the proper purchase order; verify quantities; and make a cursory examination for shipping damage. The material then shall be delivered to a Receiving Inspection area, accompanied by the purchase order receiver and all test reports and certifications which were received with the material. In-Process Inspection shall:

1. Assign lot numbers to the materials, per QMP No. 4-3.

2. Assign container numbers, per QMP No. 4-3. If the supplier has assigned container numbers, those numbers shall be recorded rather than assigning new numbers.

3. Fill out HOLD--AWAITING INSPECTION, or RESTRICTED USE tags (see Attachment A), as directed by the applicable QIP and attach to each container.

4. Fill out blocks 1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 13 and 14 of a Receiving Inspection Release, Form 59558 (see Attachment B), for each lot of material.

NOTE: A Receiving Inspection Release form shall contain only one purchase order number, one supplier lot number, and one WAFF lot number.

The Receiving Inspection Release, accompanied by all test reports and certifications which accompanied the material and the purchase order receiver shall be delivered to the cognizant Quality Control Engineer. In-Process Inspection shall then place the materials in an area designated for materials awaiting inspection, or in an area for pre-processing--as directed by the QIP.

5.2.2 Inspection of Purchased Raw Material

Upon receipt of the Receiving Inspection Release form, the test reports, certifications, and the purchase order receiver, Quality Engineering shall:

1. Evaluate the supplier's test reports and certifications to assure that the supplier data indicates conformance to the purchase order requirements. Any apparent discrepancies shall be resolved prior to inspection and pre-processing of the raw materials. A separate RIR, initiated by the cognizant Quality Engineer, may be used for this purpose if desired.

2. Initiate inspection, including sampling if required, of the raw materials, per the procedure established by the applicable Quality Inspection Procedure (QIP) (see QMP No. 7-2). Quality Engineering shall evaluate the results of the inspections performed to determine conformance to the purchase order requirements. The materials shall be dispositioned as stated in paragraph 5.3, below.

5.2.2.1 Inspection and Testing of Raw Material Samples

Samples received prior to, or with the lot shipment, shall be assigned lot and container numbers by In-Process Inspection, as stated in paragraph 5.2.1, above. In-Process Inspection shall also tag, isolate materials, fill out and deliver an RIR with test reports, certifications, and the purchase order receiver to Quality Engineering, as stated in paragraph 5.2.1, above. The lot shipment, upon receipt, shall be assigned lot and container numbers by In-Process Inspection, applying the same lot numbers as were assigned to the representative samples. The container numbers for the lot shipment shall be continued in numerical order from the container numbers applied to the sample containers. If the supplier has assigned container numbers, those numbers shall be recorded, rather than assigning new numbers. The lot of material shall be tagged and isolated by In-Process inspection, as stated in paragraph 5.2.1, above.

Upon receipt of the RIR form, the supplier test reports, certifications, and the purchase order receiver, Quality Engineering shall perform the actions stated in points 1 and 2 in paragraph 5.2.2, above. The materials shall be dispositioned as stated in paragraph 5.3, below.

5.3 Dispositioning Materials at Receiving Inspection

Quality Engineering shall review the inspection data required by the applicable QIP, upon receipt. When discrepancies are noted, the requirement and the actual condition shall be noted in blocks 11 and 12, respectively, of the RIR. All material that fully meets requirements may be immediately dispositioned as "Accept" in block 15 of the RIR by the Quality Engineer. The cognizant Quality and Process Engineers shall evaluate all discrepant material and reach a mutually agreed-upon disposition. The disposition shall be written in block 15 of the RIR, utilizing the Disposition Codes shown in block 17. Materials shall be returned to the supplier for REWORK/REPLACEMENT whenever the combination of schedule date and rework/replacement time allow this approach. The dispositions available for use are defined as follows:

1. Accept

   This disposition shall be used when the material fully meets the requirements or when, in the estimation of the Quality and Process Engineers, the material can be utilized in the manufacture of any elements such that the elements will meet WANL requirements.

2. Conditionally Acceptable

   This disposition is available for use when the production schedule makes it advisable to release material prior to having obtained all inspection data. This disposition cannot be used for accepting known discrepant mat material. When Operations requests release of material for which inspection is lacking, Quality Engineering shall contact Process Engineering and reach agreement on whether or not this disposition shall be utilized. A Process Engineering Signature, in addition to the Quality Engineering signature, shall be obtained in block 21 of the RIR, indicating concurrence with the disposition and acceptance of the risk involved, as stated on the RIR.

3. Accept, Restricted Use

   This disposition shall be used when, in the estimation of the Quality and Process Engineers, the material cannot be accepted for all uses but can be accepted for some restricted use. The particular restriction shall be stated in block 16 of the RIR. This disposition requires a signature by Process Engineering, as well as Quality Engineering, indicating concurrence with the disposition.

4. Rework

This disposition shall be used when, in the estimation of the Quality and Process Engineers, the discrepant material is capable of being reworked to the purchase order requirements, whether by WAFF or by the supplier. Rework instructions, as required, shall be stated in block 18 of the RIR. This disposition shall be agreed upon by the supplier, and the contact shall be made through the Purchasing Department. This disposition requires a signature by Process Engineering, as well as Quality Engineering, indicating concurrence with the disposition.

5. Scrap

This disposition shall be used when, in the estimation of the Quality and Process Engineers: (1) the material cannot be reworked to the purchase order requirements, or does not justify the time and expense of rework, and (2) the material has no other applications. The supplier shall be contacted, via Purchasing, to obtain concurrence for scrap dispositions. This disposition requires a signature by Process Engineering, as well as Quality Engineering, indicating concurrence with the disposition.

6. Other

This disposition may be used whenever the five dispositions defined above do not adequately define the desired processing/actions. When this disposition is used, it shall be explained in block 18 of the RIR. This disposition requires a signature by Process Engineering, as well as Quality Engineering, indicating concurrence with the disposition.

5.4 RIR Distribution Following Dispositioning

Following Engineering dispositioning, sufficient copies shall be made from the master and distributed by Quality Engineering to fill the distribution establish establish . by Quality Engineering.

5.5 Actions Required Following Distribution of the RIR

The actions required following distribution of the RIR are stated below, headed as: (1) the department receiving the copy, and (2) the type of disposition.

5.5.1 Quality Engineering (All Dispositions)

Quality Engineering shall review all RIR's and prepare Requests for Corrective Action (RCA's) as deemed necessary (see QMP No. 5-4). In addition, requests for "L" order shall be prepared and submitted to Purchasing when the RIR disposition requires that the material be returned to the supplier.

5.5.2 Receiving Inspection

5.5.2.1 Accept, Conditionally Acceptable, Accept for Restricted Use

The In-Process Inspector at Receiving Inspection shall fill out ACCEPT tags and apply to all lot containers (see Attachment A for sample tag). The material shall then be released to Operations.

5.5.2.2 Rework, Scrap

The In-Process Inspector at Receiving Inspection shall fill out REJECT tags and apply to all lot containers (see Attachment A for sample tag). The material shall then be directed to the cognizant department for reworking or scrapping.

5.5.3 Purchasing (All Dispositions)

Purchasing shall: (1) transmit all Requests for Corrective Action submitted to Quality Engineering to the cognizant supplier; (2) process all "L" orders submitted by Quality Engineering to assure that the material is returned to the supplier; (3) backcharge the suppliers as required; and (4) initiate, through the supplier, replacement of lots and material that have been dispositioned as SCRAP.

5.6 Receipt and Inspection of Purchased Tools and Gages

Tools and Gages shall be received by Receiving in the same manner as described in paragraph 5.2.1, above. Receiving Inspection of the tools and gages shall be performed per the procedure established in QMP No. 9-2.

5.7 Receipt and Inspection of Purchased Fuel

Fuel shall be received, inspected, and controlled in the same manner as stated in paragraphs 5.1 through 5.5, with the following exceptions:

1. Accountability shall perform the receiving function and shall assign lot and container numbers in a manner acceptable to Quality Engineering.
2. Accountability shall store and control the fuel. This is necessary because of the hazards involved.

NOTE: All tags shall be applied by the In-Process Inspector to the innermost metal containers to assure that the tag will not become separated from the material.

5.8 Materials Unsuitable for Core Use

All materials which are ordered for other than core use shall be tagged by In-Process Inspection with a NOT FOR CORE USE tag and released to the concerned department.

5.9 Pre-Processing of Materials

Materials requiring pre-processing operations (cross-blending, sifting, etc.) prior to combining with other raw materials shall be controlled through the pre-processing operations by the use of the standard tags (see Attachment A) and data recording forms.

[redacted]
Atomic Energy Act - 1954

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Astronuclear Laboratory

APPENDIX B

WNCO Operating Procedure, AFF 3001,
"CORE FUEL ELEMENT PROCESS AND MATERIAL
QUALIFICATION PROCEDURES"

[redacted]
Atomic Energy Act - 1954

[redacted]

[redacted]

Atomic Energy Act - 1954

Astronuclear Laboratory

[redacted]

AFF PROCEDURAL SUBMITTAL AND CHANGE

AFF PROCEDURE 3001, REVISION 0

TITLE OF THIS PROCEDURE: "CORE FUEL ELEMENT PROCESS AND MATERIAL QUALIFICATION PROCEDURES"

DATE OF THIS REVISION: OCTOBER 2, 1967

THIS REVISION SUPERSEDES AFF _____, REVISION _____

DESCRIPTION OF CHANGE:

NEW PROCEDURE.

WBS:ales

[redacted]

ENTIRE DOCUMENT CLASSIFIED IN ACCORDANCE WITH PARAGRAPH [redacted] OF CG-RR-2, [redacted] CLASSIFICATION GUIDE.

W. B. Shearer 10/2/67

W. B. Shearer, Engineer
WNCC Fabrication Engineering

[redacted]

[redacted] 10/2/67

Authorized Classifier Date

Group [redacted] Automatic Downgrading [redacted]

[redacted]

Atomic Energy

CORE FUEL ELEMENT PROCESS AND MATERIAL QUALIFICATION PROCEDURES

1.0 INTRODUCTION

Procedures used to qualify raw materials and processes for manufacturing fuel elements shall be selected to provide the data necessary to assure a high yield of elements which meet the product specification. The work scope will vary depending upon material and process changes or product-requirement changes being evaluated. Thus, changes having a major effect on the product properties should be evaluated in detail, while changes that would not significantly alter the product should not be studied. It is the manufacturer's responsibility to determine and perform the tests required to assure satisfactory product results. Qualification procedure guidelines, based on current technology, for various procurements and process changes are herein summarized. Specific detailed procedures for material procurements and process changes will be defined on work orders issued by Process Engineering.

2.0 GRAPHITE FLOUR

2.1 Procurement

Material shall conform to Purchase Department Specification 30046-C, modified as required to meet changing product requirements or to locate a supplier.

2.2 Quality Evaluation Tests

Material quality evaluation shall include the following:

(A) WNCO surveillance of the manufacturer.

(B) Evaluation of a representative sample for conformance to Purchase Order requirements.

(C) Characterization studies of representative samples for information purposes. Spectographic analysis for contaminants.

(D) Fuel element processing tests to establish manufacturing procedures for producing core quality elements.

2.3 Fuel Element Processing

Process batches of non-fueled and fueled elements through the first machining inspection operation concurrent with batches of approved core material. The objectives of these tests will be:

(A) To correlate processing characteristics of new material with previously acceptable materials, and

(B) To establish process procedures that will yield elements meeting specification requirements.

Processing should be consistent with current manufacturing procedures or procedures to be utilized in fabricating elements with this material.

The test program should provide the following data:

(A) Processing characteristics of new versus standard material, i.e., dimensional data, density and extrusion data versus binder content,

(B) Fuel element conformance to matrix inspection requirements, i.e., eddy current, visual, gamma count, body leakage and hole location,

(C) Fuel element conformance to thermal expansion and flexural strength requirements.

If significant differences exist between standard and new materials, detailed testing to determine effects of these differences on element properties and performance shall be performed.

3.0 CARBON BLACK

3.1 Procurement

Material shall conform to Purchase Department Specification 30047-B except for acceptable deviations.

...Energy Act - 1954

3.2 Quality Evaluation Tests

Tests shall include the following:

(A) Evaluation of representative sample for conformance to Purchase Order requirements.

(B) Spectrographic analysis for contaminants

(C) Fuel element processing tests to establish fabrication characteristics.

3.3 Fuel Element Processing

Process batches of non-fueled and/or fueled elements containing new material through the first machining inspection operation together with elements manufactured from approved core materials to correlate processing characteristics of new material with previously acceptable material. If processing and/or quality evaluation tests show material is significantly different from currently used material, detailed tests to show that material will yield elements meeting product requirements are necessary.

4.0 FUEL BEADS

4.1 Procurements

Material shall conform to Purchase Department Specification 30050-E except for accepted deviations.

4.2 Quality Evaluation Tests

Tests shall include the following:

(A) Evaluation of representative sample(s) for conformance to purchase order requirements. Determination of uranium and density for batch calculation purposes.

(B) Fuel element processing tests to establish uranium loadings and fabrication characteristics.

4.3 Fuel Element Processing

Process batches of fueled elements containing new material together with elements manufactured from approved core materials through the leach inspection operation. Correlate inspection and processing data of the new procurement with the standard material. If significant differences exist, perform detailed tests and the development efforts required to produce elements meeting specification requirements. Elements that have been processed and evaluated per standard operating procedures can be further processed and submitted as core-candidate elements.

5.0 VARCUM

5.1 Procurement

Material shall conform to Purchase Department Specification 30048-B except for accepted deviations.

5.2 Quality Evaluation Tests

Tests shall include the following:

(A) Evaluation of representative sample(s) for conformance to purchase order requirements.

(B) Fuel element processing tests to establish coking values and suitability of material for core elements.

5.3 Fuel Element Processing

Process batches of non-fuel elements containing new material with elements containing standard material through the first machining inspection operation. Determine coking values

of new and standard material for batch calculation purposes. Corelate inspection and processing data of the new procurement with the standard material. If significant differences exist, perform detailed tests and development efforts required to produce elements meeting specification requirements.

6.0 NIOBIUM PENTACHLORIDE

6.1 Procurement

Material shall conform to Purchase Department Specification 30052 except for acceptable deviations.

6.2 Quality Evaluation Tests

Tests shall include the following:

(A) Evaluation of a representative sample for conformance to Purchase Order requirements.

(B) The coating and evaluation of fuel elements to establish suitability of material for core elements.

6.3 Fuel Element Processing

Process leached fuel-bearing elements through the NbC coating, Mo coating and final inspection operations. The NbC coating run(s) may be of any type for which future use of this material is planned (one-step, Bore/O.D., etc.). Regular core elements may be processed along with test elements. Pending acceptance of the material, all elements coated that meet core element inspection and test evaluation requirements shall be considered core candidates.

Hydrogen corrosion test elements shall be Mo coated per standard AFF Procedure and evaluated per P.S. 294613. Acceptance shall be based on an evaluation of results by WNCO and the Fuel Evaluation Group at WANL. If elements

manufactured with the new procurement exhibit poor corrosion behavior, the following action shall be taken:

(A) If the new material is not responsible for failure, it shall be released for core use.

(B) Material deemed responsible for failure is subject to rejection.

(C) Additional test batches shall be processed if additional evaluation data are required.

7.0 MOLYBDENUM HEXACARBONYL

7.1 Procurement

Material shall conform to Purchase Department Specification 30095 except for acceptable deviations.

7.2 Quality Evaluation Tests

Tests shall include the following:

(A) Evaluation of a representative sample for conformance to Purchase Order requirements,

(B) Fuel element processing and evaluation tests to establish suitability of material for coating core elements.

7.3 Fuel Element Processing

Process NbC coated fuel-bearing elements through the Mo coating operation using Mo $(CO)_6$ of the new procurement. Regular production elements may be processed along with the test elements and submitted as core candidates if materials prove satisfactory. Perform the standard final inspection operations on test elements. Submit for hydrogen corrosion test evaluation (PS 294613).

Processing data and test results shall be evaluated by WMCO and the Fuel Evaluation Group at WANL. If elements manufactured with the new procurement exhibit poor corrosion behavior, the following action shall be taken:

(A) If the new material is not responsible for failure it shall be released for core use.

(B) Material deemed responsible for failure is subject to rejection.

(C) Additional test elements shall be processed if additional evaluation data are required.

8.0 MATRIX FABRICATION QUALIFICATION

8.1 Application

Pre-qualification tests shall be conducted on process or equipment changes that could significantly alter the quality of the fuel element matrix or the yield of acceptable elements obtained.

8.2 Qualification Tests

The test work scope shall include processing a limited number of elements to proposed changes. The effects of these changes shall be evaluated. Elements that meet all product requirements may be submitted as core candidates. Examples of tests that may be required include:

(1) Processing to establish fuel loadings or answer chemistry questions.

(2) Tests to check-out equipment modifications or new equipment.

(3) Tests to check-out material and procedural changes.

(4) Tests to evaluate effects of process deviation on product results.

9.0 LEACHING PROCESS QUALIFICATION

9.1 Application

Tests shall be conducted on leaching process or equipment changes that could significantly alter fuel element quality.

9.2 Qualification Tests

The work scope shall include processing fuel-containing elements to proposed changes and evaluating the effects of these changes on fuel element quality. The test elements shall be processed through the NbC coating and Mo coating operations. All standard non-destructive inspection operations shall be performed on the test elements. Coated elements shall be evaluated with hydrogen corrosion tests (P.S. 294613). Processing, inspection and corrosion test results shall be correlated with results obtained on elements leached by approved procedures. Elements in the qualification batches that meet all product requirements may be submitted as core candidates.

10.0 NIOBIUM CARBIDE COATING PROCESS QUALIFICATION

10.1 Application

Qualification tests shall be performed to evaluate effects of coating process parameter or equipment changes that could significantly affect fuel element quality or the yield of acceptable core elements.

10.2 Qualification Tests

Niobium carbide coating runs shall be made with fuel-containing elements using proposed process, fixturing and/or equipment changes. Coated elements shall be processed through the Mo coating operation. All standard leaching, NbC coating and Mo coating inspection operations shall be performed and evaluated. Fuel element evaluations shall include hydrogen corrosion testing per P.S. 294613.

[redacted]
Atomic Energy Act - 1954

Results shall be correlated with the corrosion test performance of standard elements. Proposed changes can be adopted if results obtained indicate the process will yield elements meeting all product requirements. Elements in the qualification runs that meet all product requirements may be submitted as core candidates.

11.0 MOLYBDENUM COATING PROCESS QUALIFICATION

11.1 Application

Qualification tests shall be performed to evaluate effects of coating process parameter, equipment and/or product requirement changes that could significantly affect fuel-element quality or the yield of acceptable core elements.

11.2 Qualification Tests

Process standard NbC O.D. coated and/or NbC one-step coated fuel containing elements with proposed Mo coating process changes. Process standard NbC coated fuel containing elements from the same O.D. and/or one-step coating runs per standard AFF Procedures. Perform standard inspection operations for NbC coated and Mo coated elements Perform hydrogen corrosion tests (P.S. 294613) on elements coated with the proposed Mo coating operation and the standard operation. Review processing data, non-destructive test results and hydrogen corrosion test results. Proposed changes can be adopted if results indicate proposed process changes will produce elements meeting product requirements.

Qualification run fuel-elements that meet all product requirements may be submitted as core candidates.

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APPENDIX C

Purchasing Department Specifications Used to Procure NERVA Raw Materials

CONFIDENTIAL
RESTRICTED DATA

INFORMATION CATEGORY
UNCLASSIFIED
Authorized Classifier Date

Westinghouse Electric Corporation

Astronuclear Laboratory
P. O. Box 10864
Pittsburgh, Pa. 15236
(Fed. Ident. Code No. 14683)

PURCHASING DEPARTMENT SPECIFICATION 30046-C
(Not for Publication)

July 19, 1967

GRAPHITE FLOUR, SECONDARY

1. SCOPE

This specification covers the requirements for secondary graphite flour of high purity, high density, and high degree of isotropy, designated as follows:

PDS Spec Designation	Description
30046-1	Isotropic, secondary graphite flour

2. APPLICABLE DOCUMENTS: None

3. REQUIREMENTS

3.1 PRIMARY RAW MATERIALS

3.1.1 Isotropic, delayed petroleum coke which has been calcined and milled into suitable particles and flour shall be used.

3.1.2 A suitable coal tar pitch shall be used as a binder for the particles of Section 3.1.1.

3.2 PROCESS OF STOCK

3.2.1 A mixture of the materials of Section 3.1 shall be extruded into suitable shape (called stock) not exceeding 50 sq. in. in cross sectional area.

3.2.2 The stock shall be temperature processed to a minimum 2700°C final temperature based upon the manufacturers standard procedure for temperature determination.

3.2.3 The stock shall be subjected to one impregnation with coal tar pitch or tar at any suitable point in the process before final graphitization.

3.2.4 The stock shall then be ground into secondary graphite flour.

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POP/cas

W form 59504A

3.3 STOCK PROPERTIES

3.3.1 The stock, after final graphitization, shall have a minimum bulk density of 1.65 gm/cc when tested as specified in Sections 4.4.5 and 4.5.3.

3.3.2 The anistropy of the stock shall be determined by measuring the specific resistance at room temperature. The ratio of specific resistance perpendicular to the extrusion direction to that parallel to the extrusion direction shall not exceed 1.20. Sample selection and techniques shall be as specified in Sections 4.4.6 and 4.5.3.

3.4 SECONDARY GRAPHITE POWDER PROPERTIES

3.4.1 Physical Properties

3.4.1.1 Particle size distribution: When tested according to Sections 4.4.4 and 4.5.4, the distribution of particle sizes shall be as follows:

Mesh Size	% By Weight
+60	0
-60+65	0-2
-65+100	4-6
-100+150	13-17
-150+200	19-23
-200+270	10-14
-270+325	7-11
-325	34-38

If necessary to blend specific sieve fractions to achieve this distribution, supplier must submit procedure in writing for Purchaser approval.

3.4.1.2 The secondary graphite powder shall be isotropic as demonstrated in the following manner: 1) Photomicrographs of samples conforming to Sections 4.4.1 and 4.5.4 shall resemble Exhibit A. An example of unacceptable material is Exhibit B. 2) The average of three determinations of the crystallographic interlayer spacing shall be between 3.361 and 3.363 Å. Sample selection and techniques employed must conform to Sections 4.4.2 and 4.5.4.

3.4.1.3 The moisture content as determined by weight loss at 110°C shall not exceed 0.2 weight %.

3.4.1.4 The average of 4 specific surface area measurements shall be between 4.0 and 6.0 m^2/gr. Sample selection and the techniques employed must conform to Sections 4.4.3 and 4.5.4.

3.4.2 Chemical Properties: When tested according to Sections 4.4.5 and 4.5.4, the flour shall conform to the following listing of average impurity maximums.

Ash	300 ppm
Iron	100 ppm
Silicon	150 ppm
Boron	3 ppm

4. QUALITY ASSURANCE

4.1 SOURCE INSPECTION

4.1.1 Material covered by this specification is subject to source surveillance by a Westinghouse Representative. This may include surveillance of the product and of the manufacturer's systems, procedures, and facilities which relate to milling, testing, and inspection of this material. The manufacturer shall perform tests and supply data as required to illustrate compliance with all requirements of this specification.

4.1.2 Material accepted by the purchaser's inspector at the place of manufacture which is later found to contain imperfections not detected at the place of manufacture, or which subsequent tests or analysis show to be not in accordance with this specification, is subject to rejection within 180 days after receipt of the qualification drum.

4.1.3 To assure implementation of Section 4.1.1 surveillance, the manufacturer shall give the Purchaser 3 days' notice of the beginning of milling operations.

4.2 CONSISTENT QUALITY

No change shall be made in the quality of successive shipments of material furnished under this specification without first obtaining the approval of the Purchaser.

4.3 QUALIFICATION SAMPLES

4.3.1 Prior to shipment of each 25,000 lb. lot, the manufacturer shall furnish the Purchaser with one 55-gallon drum containing a representative sample of the lot. One of the copies of the Section 4.6 report shall accompany the drum. The representative sample shall be made up of grab samples of equal size taken from the stream as it fills each drum in the lot. A sample shall be taken when each drum in the production lot is approximately 1/3 full. The grab samples shall be sized so that all the samples from the lot occupy between half and two-thirds of the (55-gallon) qualification sample drum.

4.3.2 The Manufacturer shall hold the lot represented by the sample described in 4.3.1 until notified by the Purchaser as to its disposition or for a maximum of 180 days after receipt of the qualification drum.

4.4 PHYSICAL AND CHEMICAL ANALYSIS

4.4.1 Photomicrographs of appropriate particulate samples shall be produced by the procedure outlined in Procedure I of the appendix.

4.4.2 Interlayer spacings of appropriate particulate samples shall be determined by the procedure outlined in Procedure II of the appendix.

4.4.3 Specific surface area of secondary graphite flour which has a particle size distribution representative of the lot shall be determined by the BET method using nitrogen absorbed at 77°K.

4.4.4 Particle size distribution shall be determined by successively sieving a 50-gram sample through the series of Tyler standard series sieves listed in 3.4.1.1.

4.4.5 Bulk density shall be determined by Procedure III of the appendix.

4.4.6 Specific resistance measurements shall be made by Procedure IV of the appendix.

4.4.7 Chemical Properties: Ash content shall be determined by weighing, suitably igniting, and re-weighing a sample of appropriate weight. Iron, silicon, and boron shall be determined by the emission spectroscopy of an ashed sample or by wet chemical analysis. In either case, the vendor shall submit a copy of his procedure to the Purchaser for approval.

4.5 SAMPLING PLAN

4.5.1 Specimens for photomicrographs: For the photomicrographs specified in Section 3.4.1.2, at least 3 samples representative of the powder lot shall be taken for mounting.

4.5.2 Specimens for interlayer spacing determinations: For the measurements required in Section 3.4.1.2, at least 3 powder samples representing the lot shall be mounted for analysis.

4.5.3 Stock density and anisotropy specimens: One sample in each direction (parallel to extrusion and perpendicular to extrusion) shall be cut from a minimum of 100 pieces of stock per 25,000-pound lot (total 200 samples), first for density as specified in Section 3.3.1 and then for anisotropy as specified in Section 3.3.2.

4.5.4 Lot Size: A lot shall consist of a maximum of 25,000 pounds prepared for processing or processed under essentially the same conditions of manufacture and time. The tests required in Section 3.4 shall be made on representative samples of the material in the qualification drum.

4.6 REPORTS: The Manufacturer shall furnish five copies of the certified test report showing the results of the tests specified in Sections 3.3 and 3.4, and bearing a statement that the material conforms to the requirements of this specification. The report shall show the Purchase Order Number; Specification Number including dash number; Lot and Drum Serial Numbers; Date of Test; Name of Manufacturer.

5. PREPARATION FOR DELIVERY

5.1 PACKING: The materials shall be shipped in 55-gallon steel drums which are clean, plastic-lined, and sealed.

5.2 MARKING: Each drum shall be plainly marked with the Purchase Order Number; Name of Manufacturer; Manufacturer's Lot Number; Drum Serial Number; PD Specification 30046 (and revision letter); Gross, Tare, and Net Weight.

APPENDIX - PROCEDURES

PROCEDURE I

PREPARATION OF PHOTOMICROGRAPHS

The following procedure shall be followed in producing photomicrographs for Section 3.4.1.2:

(a) Mount a graphite flour sample using a suitable mounting material. It is suggested that either 3/4-or 1-inch diameter cups be used and enough powder to fill about 0.1-inch depth be mounted.

(b) Evacuate to less than 26 inches of mercury before curing. Cure at suitable conditions for the mounting material.

(c) Grind the mount flat radially, cutting through the layer of graphite powder. A suggested grinding medium is 600 grit silicon carbide paper.

(d) Polish to a scratch-free surface. A suggested procedure is as follows: (1) rough polish on silk cloth using a slurry of 30 grams Linde B in 200 milliliters of 3% chromic acid. Use a fast wheel and medium pressure. (2) final polish on micro cloth, using jewelers rouge (30 grams jewelers rouge in 250 milliliters of 3% chromic acid). Use a fast wheel with medium pressure. (3) Over-polishing tends to round particles; only a short polishing time is necessary.

(e) Photograph at least 3 representative groups of mount particles on 4" x 5" or larger paper at 200X magnification.

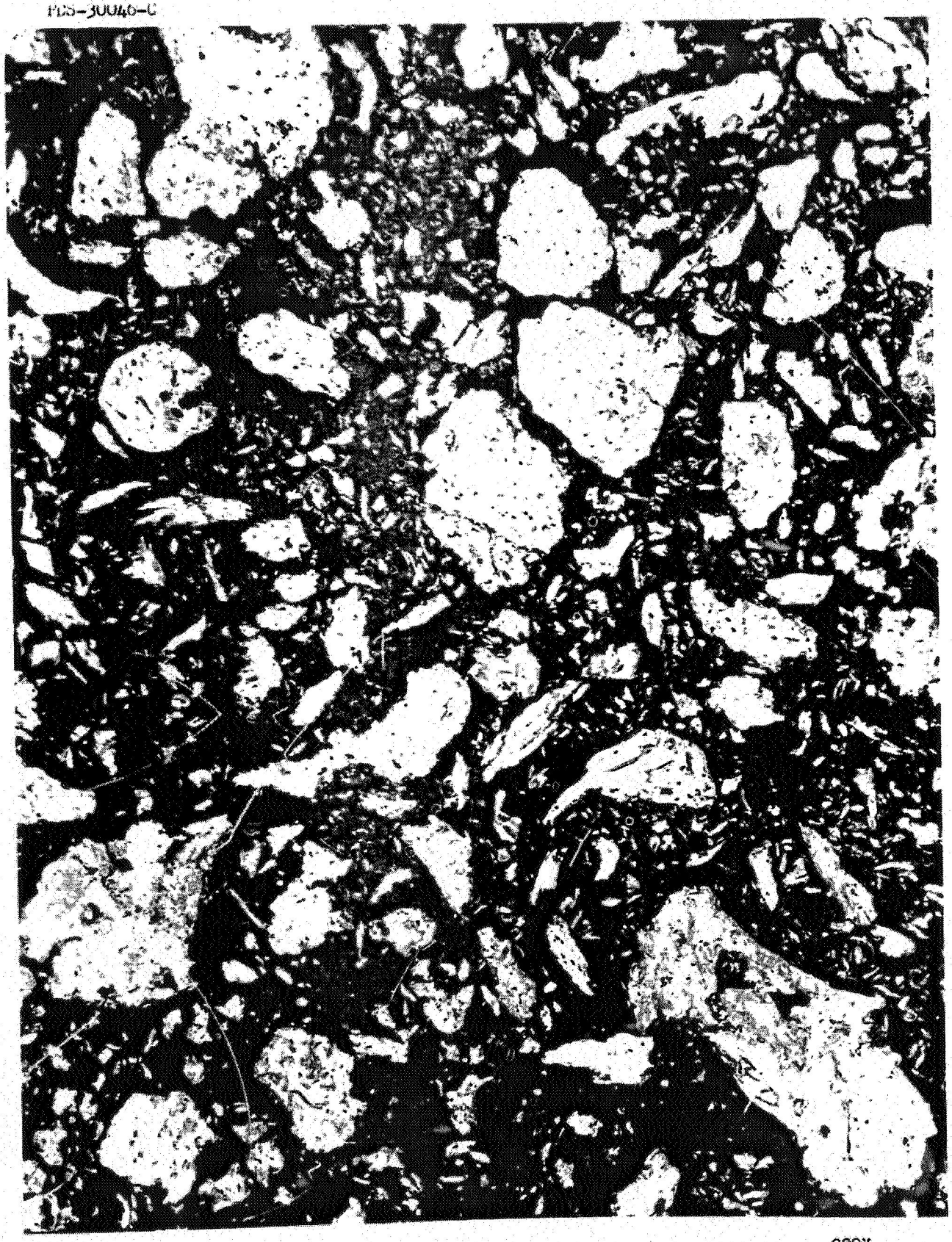

EXHIBIT A
EXAMPLE OF ACCEPTABLE ISOTROPIC SECONDARY GRAPHITE STRUCTURE

200X

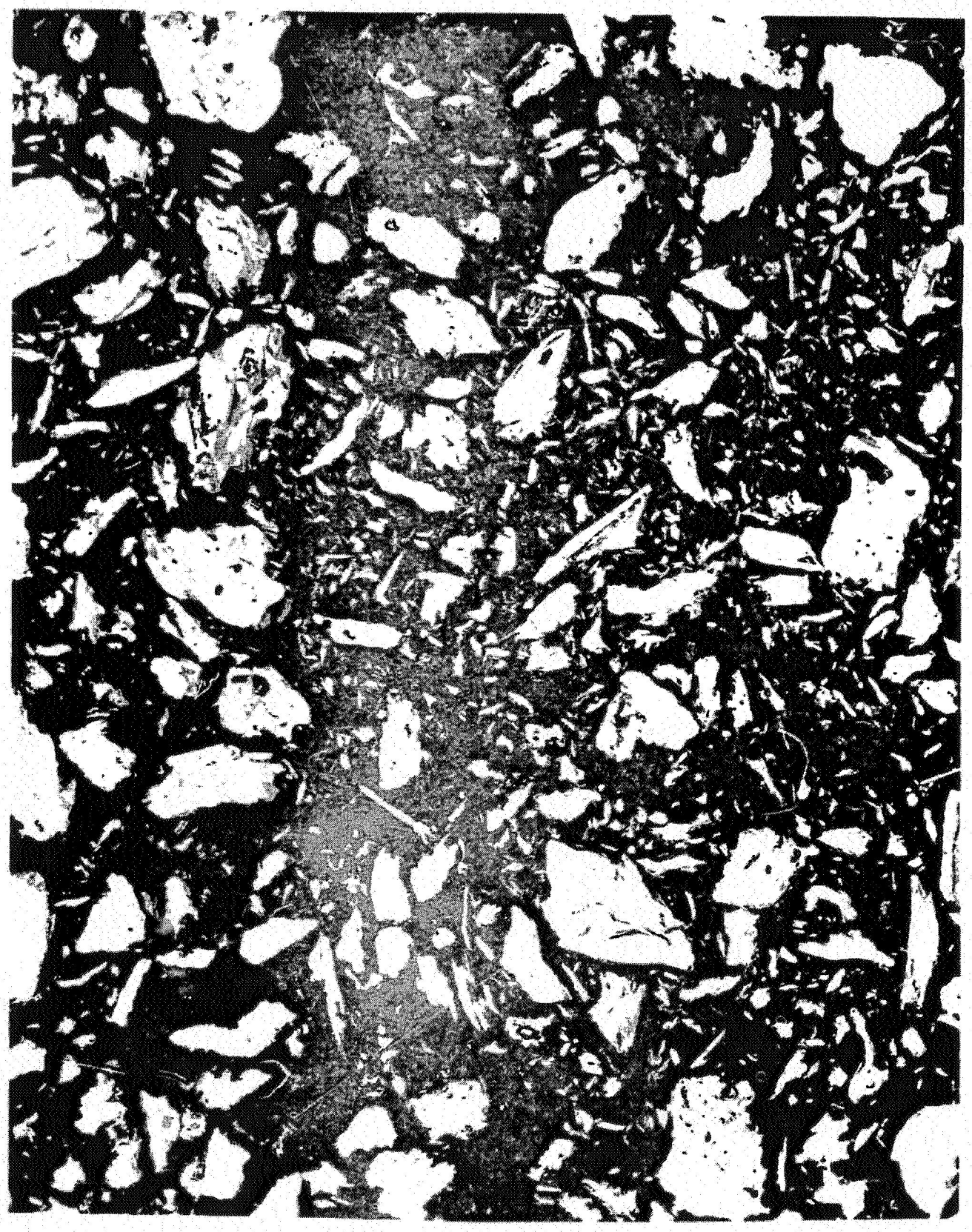

EXHIBIT B

200X

EXAMPLE OF UNACCEPTABLE ANISOTROPIC SECONDARY GRAPHITE STRUCTURE

PROCEDURE II

DETERMINATION OF CRYSTALLOGRAPHIC INTERLAYER SPACING

The following procedure shall be followed in obtaining data and calculating the interlayer spacing for Section 3.4.1.2:

(a) Sample Preparation

(1) Use a graphite sample which is representative of the lot of material in particle size distribution.

(2) Use chemically pure NaCl which has been screened through 200, on 325 mesh screens.

(3) Make an intimate mixture of 20 W/O NaCl with 80 W/O graphite sample.

(4) Mount an approximate 1-mm thick x 15-mm NaCl-graphite sample on a suitable holder. It is suggested that Fisher-Littman glass micro slides with 0.025 inch well (Fisher No. 12-550) be used. In this case the well should be filled. A suggested mounting adhesive is a 20% solution of collodion (24% alcohol) in pentyl acetate.

(b) Data Collection

(1) Copper Kα radiation shall be used.

(2) The X-ray diffraction pattern shall be determined over the range 53° to 57° (2θ) using the following settings of an appropriate diffractometer:

I. Goniometer Scanning Speed - 1/4° (2θ)/min.

II. Chart Speed - 1 in/min. or 2.5 cm/min.

The tube voltage and current settings are adjusted to attain the highest peak-to-background ratio consistent with keeping both peaks (004 of carbon and 222 of NaCl) on the chart. Slits should be adjusted to attain good resolution but the peak intensities should be near full chart width. Time constant should be adjusted to give a relatively smooth curve but be consistant with the accepted good practice of keeping it less than half the time-width of the receiving slit.

(c) Data Interpretation

(1) The angular position of each peak (004 of carbon and 222 of NaCl) shall be interpreted as the mid-point of the peak width at half peak height.

'2) The sample temperature shall be recorded and the true angular position of the NaCl 222 peak be determined at that temperature using a sodium chloride lattice constant of 2.8200 Å at 25°C(1) with a coefficient of thermal expansion of 4.02×10^{-5}/°C.

(3) The absolute angular peak position of the carbon 004 peak shall be corrected by the amount of error indicated in 2.

(4) The interlayer spacing of carbon at sample temperature shall be calculated from the peak position determined in using Braggs law and a weighted copper K α wave length of 1.54178 Å. coefficient of thermal expansion of 28.2×10^{-6}/°C.

(1) Walker, P. L, McKinstry, H. A., and Pustinger, J.V., I. & E. Chem., 8, 1651-58 (1954)

PROCEDURE III

DETERMINATION OF BULK DENSITY

The following procedure shall be used for determination of bulk density for Section 3.3.1:

(a) Sample

The material shall be cut into a circular or rectangular cross-section not smaller than 2 cm in the minor dimension and not shorter than 6.5 x the major cross-section dimension. Sides and ends must be essentially parallel with no cutting flaw to exceed 3% of the smallest dimension in depth. This sample should also be used for the determination of specific resistance in accordance with Procedure IV.

(b) Density Determination

The cross-sectional dimensions and length shall be measured to the nearest 0.01 cm. The weight shall be determined to the nearest 0.01 gram. The density is determined by dividing the weight by the volume of the sample.

PROCEDURE IV

DETERMINATION OF SPECIFIC RESISTANCE

The following procedure shall be used for determination of specific resistance for Section 3.3.2:

(a) Sample

The material shall be cut into a circular or rectangular cross-section not smaller than 2 cm in the minor dimension and not shorter than 6.5 x the major cross-sectional dimension. Sides and ends must be essentially parallel with no cutting flaw to exceed 3% of the smallest dimension in depth.

(b) Specific Resistance

A suitable direct current shall be passed through the long dimension of the sample. The potential drop over a measured length of sample shall be determined with minimal current flow through the potential probes and with neither probe closer than three centimeters to a current contact as measured along the length of the sample. The specific resistance is calculated as follows:

$$S.R. = \frac{EC}{IL}$$

Where:

S.R. = specific resistance, ohm - cm
E = potential drop, volts
C = cross-sectional area, cm^2
I = current, amps
L = length between voltage probes, cm

INFORMATION CATEGORY
UNCLASSIFIED
Authorized Classifier Date

Westinghouse Electric Corporation
Astronuclear Laboratory
P. O. Box 10864
Pittsburgh, Pa. 15236
(Fed. Ident. Code No. 14683)

PURCHASING DEPARTMENT SPECIFICATION 30047-B
(Not for Publication)

December 4, 1964

CARBON BLACK

1. SCOPE

This specification covers the requirements for high-purity carbon black, designated as follows:

P D Spec Designation	Description
30047-1	High-purity carbon black made from natural gas by the medium thermal (MT) process

2. APPLICABLE DOCUMENTS: None

3. REQUIREMENTS

3.1 PROCESS: The carbon black shall be made from natural gas by the medium thermal (MT) process.

3.2 CHEMICAL PROPERTIES: The chemical composition of the material shall not exceed the following impurity limits:

Impurity	Limit, ppm
Ash	2000
Iron	100
Silicon	500
Boron	1

3.3 PHYSICAL PROPERTIES

3.3.1 Particle Size: Not more than 0.10% by weight of the tested sample shall remain on the screen of a U.S. Standard Sieve No. 325 (44 microns).

3.3.2 Particle Density: The particle density shall be 1.85 $\pm$ 0.10 gm/cc.

3.3.3 Moisture Content: The moisture content shall not exceed 0.5% of the initial sample weight.

3.3.4 High-Density Inclusions: A one-kg sample of the material shall contain no more than 25 high-density inclusions. Of these 25 permissible, no more than ten may be larger than 0.010-inch diameter.

4. QUALITY ASSURANCE

4.1 SOURCE SURVEILLANCE

4.1.1 Material covered by this specification is subject to source surveillance by a quality representative of the purchaser. This may include surveillance of the product and of the manufacturer's systems, procedures, and facilities which relate to the manufacture and inspection of this material. The manufacturer shall perform tests and supply data at no extra cost, as required to illustrate compliance will all requirements of this specification.

4.1.2 Material accepted by the purchaser's inspector at the place of manufacture which is later found to contain imperfections not detected at the place of manufacture, or which subsequent tests or analysis show to be not in accordance with this specification, is subject to rejection.

4.2 CONSISTENT QUALITY: No change shall be made in the quality of successive shipments of material furnished under this specification without first obtaining the approval of the purchaser.

4.3 QUALIFICATION

4.3.1 Prior to shipment of each lot, the manufacturer shall furnish the purchaser with twenty 50-pound bags of material taken at random from a group of 80 which constitute a series of bags consecutively filled during a production run. The 20-bag sample shall be accompanied by the test report referenced in Section 4.5, and identifying the bags with the lot number assigned to the 80-bag group.

4.3.2 The manufacturer shall hold the remaining 60 bags of the group until notified by the purchaser as to its disposition.

4.3.3 Notification of disposition shall be as follows:

4.3.3.1 If the materials conforms to Section 3, the 60 remaining bags shall be released for shipment to the purchaser.

4.3.3.2 If the material does not conform to Section 3, the 60 bags remaining shall be released for manufacturer's other disposition.

4.4 TESTING

4.4.1 Test procedures and sampling shall be designated by the purchaser at time of invitation for bids.

4.4.2 The impurity content shall be determined by emission spectroscopy or other equivalent methods.

4.5 REPORTS: The manufacturer shall furnish five copies of a certified test report showing the results of the tests specified in Section 4.4, and bearing a statement that the material conforms to the requirements of this specification. The report shall show the Purchase Order Number: Specification Number, including dash number; Test Identification Number: Date of Test: Name of Manufacturer.

5. PREPARATION FOR DELIVERY

5.1 PACKAGING

5.1.1 All shipping containers shall be of the moisture-resistant type.

5.1.2 Each bag shall contain not less than 50 pounds of carbon black.

5.2 MARKING: Each bag shall be plainly marked with the Purchase Order Number; Manufacturer's Lot Number; PDS 30047 (including revision letter); and Name of Manufacturer.

Westinghouse Electric Corporation
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Pittsburgh, Pa. 16236
(Fed. Ident. Code No. 14683)

PURCHASING DEPARTMENT SPECIFICATION 30048-B December 4, 1964
(Not for Publication)

RESIN, FURFURYL ALCOHOL

1. SCOPE

This specification covers the requirements for furfuryl-alcohol resin, designated as follows:

P D Spec Designation	Description
30048-1	Partially polymerized furfuryl-alcohol resin

2. APPLICABLE DOCUMENTS - None

3. REQUIREMENTS

3.1 PROCESS: This material shall be made from furfuryl alcohol.

3.2 CLEANLINESS: All handling, processing, and packaging shall be adequately controlled to prevent contamination of the material. Boron, boron-containing compounds, or boron-contaminated equipment shall not be used in processing this material.

3.3 CHEMICAL AND PHYSICAL PROPERTIES:

3.3.1 Impurities: The resin shall contain less than two ppm boron.

3.3.2 Resin Solids: The resin-solids content shall be 54% ± 1%.

3.3.3 Viscosity: The viscosity shall be 250 cps ± 15% at 25°C.

3.3.4 Acidity: The pH of the resin shall be five to six.

3.3.5 Reactivity: The average gel time at 135°C shall be 5.0 to 6.5 minutes as calculated from three determinations with a Sunshine Gel Time Meter.

4. QUALITY ASSURANCE

4.1 SOURCE SURVEILLANCE

4.1.1 Material covered by this specification is subject to source surveillance by a quality representative of the purchaser. This will include surveillance of the product and of the manufacturer's systems, procedures, and facilities which relate to the manufacture and inspection of this material. The manufacturer shall perform tests and supply data at no extra cost, as required to illustrate compliance with all requirements of this specification.

4.1.2 Material accepted by the purchaser's inspector at the place of manufacture which is later found to contain imperfections not detected at the place of manufacture, or which subsequent tests or analysis show to be not in accordance with this specification, is subject to rejection.

4.2 CONSISTENT QUALITY: No change shall be made in the quality of successive shipments of material furnished under this specification without first obtaining the approval of the purchaser.

4.3 QUALIFICATION SAMPLE

4.3.1 Prior to shipment of material from a production lot, the manufacturer shall furnish to the purchaser a five-gallon sample representative of the lot.

4.3.2 Until notified by the purchaser as to the disposition of the remaining material from the lot, the manufacturer shall maintain the material at temperatures in the range 40-65°F.

4.3.3 If partial shipment is made from a lot, further shipments from that lot must be completed before shipment from another lot is begun.

4.4 TESTS

4.4.1 All test procedures to prove conformance to the requirements of Section 3.3 shall be submitted for the purchaser's written approval.

4.4.2 Reactivity: The resin shall be catalyzed by the addition of 1.0 grams of maleic anhydride to 30.0 grams of resin.

4.5 LOT SIZE: A lot shall be a maximum of 16,500 pounds of the resin, produced in a single uninterrupted production run.

4.6 REPORTS: The manufacturer shall furnish five copies of a certified test report showing the results of the tests specified in Section 4.4; and bearing a statement that the material conforms to the requirements of this

specification. The report shall show the Purchase Order Number; Specification Number, including dash number; Test Identification Number; Date of Test; Name of Manufacturer.

5. PREPARATION FOR DELIVERY

5.1 PACKING: The material shall be packed in clean, tight 55-gallon drums.

5.2 MARKING: Each drum shall be plainly marked with the Purchase Order Number; Name and Grade of Material; Name of Manufacturer; Manufacturer's Lot Number; Drum Serial Number; PDS 30048 (including revision letter); Gross, Tare, and Net Weight.

Westinghouse Electric Corporation

Aug 5, 1966

File No.
P D Spec 51300AB Rev C

(Federal CODE IDENT NO. 79500)

MALEIC ANHYDRIDE

1. This specification covers high purity maleic anhydride.

2. No change shall be made in the quality, packaging or labeling of successive shipments of material furnished under this specification without first obtaining the approval of the purchaser.

MANUFACTURE

3. MANUFACTURE: Material shall be of uniform quality in crystalline state, free from foreign matter and discoloration. Material may be in the form of powder or fused briquettes.

CHEMICAL PROPERTIES AND TESTS

4. COMPOSITION:

Maleic Anhydride Content, %, Min	99.5
Ash Content, ppm, max (by weight)	2.
Moisture Content, %, max (by weight)	0.2

PHYSICAL PROPERTIES AND TESTS

5. COLOR OF MOLTEN SAMPLE: Not greater than 20 (Hazen).

6. SOLIDIFICATION POINT: Shall be in range 52 to 54 C (125.6 to 129.2 F) inclusive.

STORAGE

7. TEMPERATURE AND HUMIDITY: Storage shall be in a dry environment at a temperature below 32.2 C (90 F).

8. STORAGE LIFE: After prolonged storage per Section 7, the maleic anhydride shall still meet the requirements of this specification.

CERTIFICATION

9. TEST REPORTS: With each shipment the supplier shall furnish to the purchaser 3 copies of a certified test report certifying that a representative sample of each lot of material used conforms to the requirements of this specification.

PACKING AND MARKING

10. PACKING: Material shall be shipped in clean, sealed containers (Max weight 5 pounds, net) as agreed between purchaser and supplier.

11. MARKING: Each carton (package of one or more containers per Section 10 as shipped) shall be plainly labeled on two sides as follows: Purchase Order Number; Maleic Anhydride, P D Spec 51300AB Rev C; Minimum Per Cent Content of Maleic Anhydride 99.5; Gross, Tare, Net Weight; Manufacturer's Lot No.; Manufacturer's Name and Plant Location.

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INFORMATION CAT[redacted]ORY
C-RD
[redacted] 3/6/67
Auth[redacted]ized Classifier: Date

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GROUP I
Excluded from [redacted]ic Down-grading [redacted]assification

PURCHASING DEPARTMENT SPECIFICATION 30050-E
(Not for Publication)

SPECIAL URANIUM PARTICLES
(Title Unclassified)

March 1, 1967

1. SCOPE

This specification covers the requirements for pyrolytic-carbon-coated uranium dicarbide (UC_2) particles, designated as follows:

PD Spec. Designation	Description
PDS 30050-1	Pyrocoated UC_2 fuel beads for elements (in specified U migration test, unmigrated coating thickness $\bar{x}$-1.60σ to be at least five microns)
PDS 30050-2	Same as -1, except (...at least 12 microns)

2. APPLICABLE DOCUMENTS

The following documents, of the issue in effect on the date of invitation for bids, shall form a part of this specification to the extent specified herein:

Handbook of Federal Regulations, USAEC Division of Construction and Supply, Traffic Management Section, Washington, D.C., May, 1958.

Nuclear Safety Guide, TID 7016 USAEC Technical Information Service Extension, Oak Ridge, Tennessee.

ASTM E-11

H. H. Gill, R. F. Rolph, and G. W. Armstrong, Analyt. Chem. Vol. 30 pp. 1788 - 1792, 1958.

Spectrophotometric Determination of U with 1-(2-Pyridylazo) - 2 Naphthol

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3. REQUIREMENTS

3.1 CAUTIONARY PROCEDURE FOR RADIOACTIVE MATERIALS: In conformance with the pertinent regulations listed in the applicable documents, (1) work with uranium-containing particles shall be controlled to prevent ingestion of radioactive materials and over-exposure to radiation; (2) criticality hazards associated with enriched fuel shall be evaluated and controlled; and (3) shipments shall be packaged to assure nuclear safety and prevent contamination.

3.2 MANUFAC...

3.2.1 Process: UC_2, fully enriched to the 93.15 ± 0.15% level, shall be manufactured into spherical particles, and the particles shall be coated with a carbon coating deposited by a pyrolytic process.

3.2.2 Raw Material: To assure conformance with the enrichment requirement of Section 3.4.1, it shall be the manufacturer's responsibility to ascertain the validity of the AEC isotopic analyses for the raw material used for manufacturing these particles. The purchase order will specify in kilograms (kg) the total quantity of contained uranium required in the coated particles to be delivered.

3.3 QUALIFICATION

3.3.1 Feasibility: To enable the purchaser to evaluate the capability of the manufacturer to meet this specification with the quantities of material and the schedules specified in the purchase order, the manufacturer shall submit a feasibility report relating his equipment and processes with the quality and quantity of material (and the delivery schedules) specified in this specification and the purchase order, and showing his capability for meeting all necessary precautions for handling these radioactive materials.

3.3.2 Process: To enable the purchaser to establish the mechanical integrity of the coated particles in the purchaser's processes, the manufacturer shall submit a qualification sample of coated UC_2 particles. Details of the sampling, accompanying analyses, and restrictions on production before and after submission of the sample are specified in Section 4.3.

...Energy Act - 1954

3.3.3 Test Procedures: The manufacturer shall submit, for the written approval of the purchaser, proposed detailed testing procedures for all properties requiring determination. Approval is not required for applicable ASTM procedures, or for those detailed in the Appendix of this specification, provided the manufacturer agrees to use these procedures.

3.4 CHEMICAL PROPERTIES

3.4.1 Enrichment: The isotopic analysis for each lot of coated UC_2 particles shall be in the range $\pm$ 0.15% from the accumulative average of the AEC-certified isotopic values for all cylinders of UF_6 delivered and used during a production run.

3.4.2 Uranium Content: The weight percent (w/o) of uranium (U) in each lot of coated particles shall conform to both of the following limits: (a) in the range 60 to 72 w/o and (b) in the range $\pm$ 2.0 w/o from the value established for the qualification sample specified in Section 3.3.2.

3.4.3 Carbon Content: Total carbon content of the coated UC_2 particles shall be determined to the nearest 0.2% by weight.

3.4.4 Impurities

3.4.4.1 The oxygen content and the nitrogen content of the coated particles shall each be less than 2000 ppm of the uranium content.

3.4.4.2 The whole coated particles shall conform to the following limits on spectrographically-determined impurities:

3.4.4.2.1 The total of all inorganic impurities shall be less than 1000 ppm of the U content.

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3.4.4.2.2 The content of individual elements in the whole coated particles shall not exceed the following ppm of the U content:

Element	Limit, ppm	Element	Limit, ppm
Silver	1	Antimony	10
Gold	10	Silicon	300
Boron	3	Thorium	30
Barium	10	Thallium	20
Cadmium	3	Vanadium	40
Iron	500	Tungsten	30
Indium	5	Samarium	0.6 total
Lithium	2	Europium	
Molybdenum	100	Gadolinium	
Nickel	80	Dysprosium	

3.5 MECHANICAL AND PHYSICAL PROPERTIES

3.5.1 Uncoated Particles: In the procedure referenced in Section 4.5.2.4, the shape of the uncoated UC_2 particles shall conform to the following:

	Required percentage of all particles, minimum	Ratio of diameter on major axis to diameter on minor axis
3.5.1.1	95	$\leq$ 1.50
3.5.1.2	98	$\leq$ 2.00

3.5.1.3 Particles which are doublets, clumps, and sharp-cornered or angular shall not (a) qualify under the quantities specified in Sections 3.5.1.1 and 3.5.1 2, nor (b) exceed a combined total of two per cent of the sample.

NOTE: The size of the uncoated particles is not specified as such; conforming to the requirements on uranium content (Section 3.4.2), density as coated (Section 3.5.2.1), and size as coated (Section 3.5.2.2) will necessitate close control on the size of the uncoated particles.

3.5.2 Coated Particles

[redacted]

3.5.2.1 Density: The coated particles shall conform to each of the following limits: (a) in the range ± 0.200 gm/cc of the density value established in the approved qualification sample specified in Section 3.3.2; (b) in the applicable range from the density table below, based on the level of U content which becomes established under the requirements of Section 3.4.2; and (c) within a total range of variation (among the individual density tests specified in Section 4.6.3.6.2) not exceeding 0.050 gm/cc.

Density Table

w/o of U in coated particles	Permissible range of density of coated particles, gm/cc
60	3.95 - 4.55
61	4.05 - 4.65
62	4.15 - 4.70
63	4.25 - 4.80
64	4.35 - 4.90
65	4.45 - 5.00
66	4.55 - 5.10
67	4.65 - 5.20
68	4.75 - 5.35
69	4.85 - 5.45
70	4.95 - 5.60
71	5.05 - 5.75
72	5.20 - 5.90

3.5.2.2 Particle Size: In the screening procedure referenced in Section 4.4.2, the coated particles shall conform to the following limits of size distribution:

Mesh Size	Weight Per Cent
+ 60	0
-60 + 65	2.0 max
-65 + 100	28 to 70
-100 + 170	28 to 70
-170 + 200	2.0 max
-200	0

[redacted]

3.5.2.3 Extraneous Matter: In the examination specified in Section 4.4.3, finished lots of coated UC_2 particles shall contain less than 35 foreign particles (such as carbon, graphite, metals, plastics, and brush bristles) per gram of sample.

3.5.2.4 Gamma Activity: In the procedure referenced in Section 4.5.2.5, the gamma radiation spectrum of the particles shall indicate no extraneous radiation as compared with a standard source spectrum.

3.5.2.5 Coating Integrity: The leaching procedure referenced in Sections 4.5.1 and 4.5.2.6 shall not leach out more than 0.2% of the U content * of the samples of coated particles specified in (a) Sections 4.5.1 and 4.6.2.1, and (b) Section 4.5.2.6.

3.5.2.6 Thermal Stability

3.5.2.6.1 In the test referenced in Section 4.5.2.7, the result λ-1.600 shall equal or exceed the following:

PDS-30050-1 5 microns
PDS-30050-2 12 microns

3.5.2.6.2 The fraction of individual coated particles which, as represented in the microradiographs referenced in Section 4.5.2.7, show U migration completely through the coating at any point, shall not exceed 3%.

* Under Section 3.5.2.5(a), an assumed U content value of 72% by weight of the sample shall be used to establish the 0.2% leaching limit. Under Section 3.5.2.5(b), the U content value used to establish the leach limit shall be that determined for the lot under Section 4.5.2.6.

4. QUALITY ASSURANCE

4.1 SOURCE SURVEILLANCE

4.1.1 Material covered by this specification is subject to source surveillance by a Quality Representative of the purchaser. This will include surveillance of the product and of the manufacturer's systems, procedures, and facilities which relate to the manufacture and inspection of this material. The manufacturer shall perform tests and supply data, at no extra cost, as required to illustrate compliance with all requirements of this specification.

4.1.2 Material accepted by the purchaser's inspector at the place of manufacture which is later found to contain imperfections not detected at the place of manufacture, or which subsequent tests or analysis show to be not in accordance with this specification, is subject to rejection.

4.2 CONSISTENT QUALITY: No change shall be made in the quality of successive shipments of material furnished under this specification without first obtaining the approval of the purchaser.

4.3 QUALIFICATION

4.3.1 The feasibility report, qualification sam[illegible] procedures, and analyses required by Section 3.3 shall be sub[illegible] by the manufacturer and approved in writing by the purch[illegible] before processing is begun on the production quantity of material to be made under this specification. The qualification sam[illegible] (Section 4.6.1) shall be made by the processes which [illegible] used to prepare the total production quantity, and shall b[illegible] esentative of what would be supplied as the production quantity. Once the qualification sample is approved for starting production, the manufacturer shall not alter any processing procedure without prior written approval of the purchaser.

Similarly, once the test procedures have been approved by the purchaser, changing them shall require written approval of the purchaser.

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4.3.2 Results and certifications from the tests and analyses specified for acceptance of the production quantity (Sections 4.8.3 and 4.8.4) shall be supplied for each qualification sample submitted, and shall be submitted no later than the time of shipment of the sample.

4.4 NON-DESTRUCTIVE TESTS

4.4.1 Conformance to Section 3.5.2.1 (density) shall be determined for each lot on the manufacturer's 50-gm test sample (specified in Section 4.6.2.2) for the lot.

4.4.1.1 Unless Section 4.4.1.2 applies, the tests shall be two separate gas displacement measurements (Section 4.6.3.6.2) of the full 50-gm sample (Section 4.6.2.2.1).

4.4.1.2 Tests by small sample displacement of a dense fluid will require (a) the written approval of the purchaser and (b) measurements, separately reported, of duplicate samples split with a riffle from the 50-gm sample (Section 4.4.1) for the lot.

4.4.2 Conformance to the requirements on particle size distribution of the coated particles (Section 3.5.2.2) shall be determined for each lot on samples split with a riffle from the manufacturer's 50-gm test sample (specified in Section 4.6.2.2) for the lot. The method for determination is specified in Section A.1 of the Appendix. The sample shall be as specified in Section 4.6.2.2.2.

4.4.3 Conformance to the Section 3.5.2.3 dross limit shall be determined for each lot on samples split with a riffle from the manufacturer's 50-gm test sample (specified in Section 4.6.2.2.) for the lot. The determination shall be by macroexamination of the sample specified in Section 4.6.2.2.3.

4.5 DESTRUCTIVE TESTS

4.5.1 Conformance to the coating integrity requirement of Section 3.5.2.5(a) shall be determined by the manufacturer for each coating batch on a sample split with a riffle from the batch before the coating batches are cross blended to form the lot. The integrity determination shall be by the chemical tests specified in Section A.2 of the Appendix.

Note: The sampling for these coating-batch tests need not be duplicated for the purchaser as is required in Section 4.6.2.2 for the lot-based tests, and the results need not be reported, but shall be retained as specified in Section 4.8.2.1.

4.5.2 Conformance to Sections 3.4.2, 3.4.3, 3.4.4, 3.5.1,* 3.5.2.4, 3.5.2.5(b), and 3.5.2.6 shall be determined for each lot on samples split with a riffle from the manufacturer's 50-gm test sample (specified in Section 4.6.2.2.3) for the lot.

4.5.2.1 The U-content analyses specified in Section 4.6.3.1 for determining conformance to Section 3.4.2 shall each be performed on a separate sample as specified in Section 4.6.2.2.4.

4.5.2.2 The carbon-content analyses specified in Section 4.6.3.2 for the determinations specified in Section 3.4.3 shall each be performed on a separate sample.

4.5.2.3 The oxygen and nitrogen analyses specified in Section 4.6.3.3 for determining conformance to Section 3.4.4.1 shall be performed on the sample(s) specified in Section 4.6.2.2.5.

4.5.2.4 Conformance to the Section 3.5.1 requirements on uncoated particle shape shall be determined by the micro-radiographic method detailed in Section A.3 of the Appendix or by other method approved in writing by the purchaser. The sample shall be as specified in Section 4.6.2.2.6.

4.5.2.5 Conformance to the Section 3.5.2.4 requirement on gamma activity shall be determined as specified in Section A.4 of the Appendix. The sample shall be as specified in Section 4.6.2.2.7.

*Section 3.5.1 may be exempted from this Section 4.5.2 requirement if, for another method approved under Section 4.5.2.4, an uncoated sample is required.

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4.5.2.6 Determination of conformance to the coating integrity requirement of Section 3.5.2.5(b) shall be performed (a) to the procedure of Section A.2 of the Appendix and (b) on material from the lot-based test sample specified in Sections 4.5.2, 4.6.2.2, and 4.6.2.2.8.

4.5.2.7 Conformance to the Section 3.5.2.6 requirements on thermal stability shall be determined by microradiography as specified in Section A.5 of the Appendix. The sample shall be as specified in Section 4.6.2.2.9.

4.6 SAMPLING

4.6.1 Qualification: The U content (a minimum of two kg) and enrichment of the qualification sample (Section 4.3.1) will be specified in the purchase order.

4.6.2 Tests on Production Material

4.6.2.1 Coating Integrity: For those coating integrity tests which are specified in Section 4.5.1, sampling shall be in the proportion 2 gm for each 1000 gm of batch for coating batches not exceeding 2.5 kg; for batches exceeding 2.5 kg, the sample shall be 5 gm.

4.6.2.2 Other Tests: For all tests required of the manufacturer other than those specified in Sections 4.6.1 and 4.6.2.1, a 50-gm test sample shall be taken from each lot, as follows: A quantity of at least 150 gm gross weight of coated particles shall be split from the lot with a riffle in such a way as to insure that typical analyses to this specification, performed on the quantity, will be representative of the entire lot. The 150-gm quantity shall then be similarly divided with a riffle into three approximately equal test samples, two for check and referee tests as specified in Section 4.9.1.4 and one for the manufacturer's 50-gm test sample.

[redacted]

Those sample sizes which are specified for the lot-based tests shall be as follows:

	Test and Ref. Sec.	Sample Size
4.6.2.2.1	Density (4.4.1.1)	50 gm[1]
4.6.2.2.2	Particle size (4.4.2)	10 gm
4.6.2.2.3	Dross count (4.4.3)	1 gm min.
4.6.2.2.4	U analysis (4.5.2.1)	10 ± 1 gm
4.6.2.2.5	O_2 and N_2 analysis (4.5.2.3)	2 gm min.
4.6.2.2.6	Particle shape (4.5.2.4)	50 mg
4.6.2.2.7	Gamma spectrum (4.5.2.5)	0.5000 gm
4.6.2.2.8	Coating integrity (4.5.2.6)	5.0 gm[2]
4.6.2.2.9	Thermal stability (4.5.2.7)	0.5 to 1.0 gm

4.6.3 Sampling and Test Summary: This section lists the sampling for each lot, together with the cross references for the requirements; the specimens, tests, and procedures; and the records and reports or certifications.

4.6.3.1 U Content: Requirement, Sec. 3.4.2

4.6.3.1.1 Specimens and Test, Sec. 4.5.2, 4.5.2.1

4.6.3.1.2 Sampling, Sec. 4.6.2.2, 4.6.2.2.4; Two analyses

4.6.3.1.3 Records and Reports, Sec. 4.8.2.2, 4.8.3, 4.8.3.3.

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[1] Manufacturer's entire lot sample (Section 4.6.2.2).

[2] or less, if applicable under Section 4.6.2.1

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4.6.3.2 C Content: Requirement (Information only), Sec. 3.4.3

4.6.3.2.1 Specimens and Test, Sec. 4.5.2, 4.5.2.2

4.6.3.2.2 Sampling, Sec. 4.6.2.2; Two analyses

4.6.3.2.3 Records and Reports, Sec. 4.8.2.2, 4.8.3, 4.8.3.4

4.6.3.3 O_2 and N_2 Limits: Requirement, Sec. 3.4.4.1

4.6.3.3.1 Specimens and Test, 4.5.2, 4.5.2.3

4.6.3.3.2 Sampling, Sec. 4.6.2.2, 4.6.2.2.5; one sample or one sample for each element

4.6.3.3.3 Records and Reports, 4.8.2.2, 4.8.3

4.6.3.4 Impurity Limits: Requirement, Sec. 3.4.4.2

4.6.3.4.1 Specimens and Test, Sec. 4.5.2

4.6.3.4.2 Sampling, Sec. 4.6.2.2; Analyze for elements listed

4.6.3.4.3 Records and Certification, Sec. 4.8.2.2, 4.8.4

4.6.3.5 Uncoated Particle Shape: Requirements, Sec. 3.5.1

4.6.3.5.1 Specimens, Test, and Procedure: Sec. 4.5.2, 4.5.2.4, A.3*

4.6.3.5.2 Sampling, Sec. 4.6.2.2, 4.6.2.2.6; One procedure

4.6.3.5.3 Records and Certification, Sec. 4.8.2.2, 4.8.4

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* Not mandatory - see Sec. 4.5.2.4

[redacted]

4.6.3.6 Density: Requirements, Sec. 3.5.2.1

4.6.3.6.1 Specimens and Test, Sec. 4.4.1

4.6.3.6.2 Sampling, Sec. 4.6.2.2, 4.6.2.2.1; Two tests on the same entire 50-gm sample

4.6.3.6.3 Records and Reports, Sec. 4.8.2.2, 4.8.3, 4.8.3.1

4.6.3.7 Coated Particle Size: Requirements, Sec. 3.5.2.2

4.6.3.7.1 Specimens, Test, and Procedure, Sec. 4.4.2, A.1

4.6.3.7.2 Sampling, Sec. 4.6.2.2, 4.6.2.2.2; One procedure

4.6.3.7.3 Records and Reports, Sec. 4.8.2.2, 4.8.3

4.6.3.8 Dross Limit: Requirement, Sec. 3.5.2.3

4.6.3.8.1 Specimens and Test, Sec. 4.4.3

4.6.3.8.2 Sampling, Sec. 4.6.2.2, 4.6.2.2.3; One examination

4.6.3.8.3 Records and Reports, Sec. 4.8.2.2, 4.8.3, 4.8.3.2

4.6.3.9 Gamma Activity: Requirement, Sec. 3.5.2.4

4.6.3.9.1 Specimens, Test, and Procedure, Sec. 4.5.2, 4.5.2.5, A.4

4.6.3.9.2 Sampling, Sec. 4.6.2.2, 4.6.2.2.7; One procedure

4.6.3.9.3 Records and Certification, Sec. 4.8.2.2, 4.8.4

4.6.3.10 Coating Integrity (Manufacturer's batch-based tests)

Requirement, Sec. 3.5.2.5(a)

4.6.3.10.1 Specimens, Test, and Procedure, Sec. 4.5.1, A.2

4.6.3.10.2 Sampling, Sec. 4.6.2.1;
One procedure per coating batch

4.6.3.10.3 Records and Certification,
4.5.1 (Note), 4.8.2.1, 4.8.4

4.6.3.11 Coating Integrity (Lot-based tests)
Requirement, Sec. 3.5.2.5(b)

4.6.3.11.1 Specimens, Test, and Procedure,
Sec. 4.5.2, 4.5.2.6, A.2

4.6.3.11.2 Sampling, Sec. 4.6.2.2, 4.6.2.2.8;
one procedure

4.6.3.11.3 Records and Reports, Sec. 4.8.2.2, 4.8.3

4.6.3.12 Thermal Stability: Requirement, Sec. 3.5.2.6

4.6.3.12.1 Specimens, Test, and Procedure,
Sec. 4.5.2, 4.5.2.7, A.5

4.6.3.12.2 Sampling, Sec. 4.6.2.2, 4.6.2.2.9;
One procedure

4.6.3.12.3 Records and Reports, Sec. 4.8.2.2, 4.8.3

4.6.4 Batch and Lot

4.6.4.1 A batch shall mean a coating batch - that quantity of particles coated together in a single coating-furnace loading.

4.6.4.2 A lot of coated particles shall be determined by the U content, which shall be in the range five to ten kg. If processing sublots are necessary, the manufacturer shall demonstrate that his cross blending of sublots homogenizes the final lot so that typical analyses to this specification, performed on any sublot, are representative of the entire final lot.

NOTE: A larger lot may be negotiated upon satisfactory conformance to this specification and demonstration of homogeneity of the larger-lot material, i.e., furnishing evidence, satisfactory to the purchaser, that typical analyses to this specification, performed on material from any shipping container from the lot, are representative of the entire final lot.

4.7 ACCEPTANCE

4.7.1 Acceptance of each lot of coated particles will be subject to tests and analyses by the purchaser for all requirements of Section 3.4 and of Section 3.5 except 3.5.2.5(a).

4.7.2 If results from purchaser tests or analyses disagree with the corresponding ones of the manufacturer, the two parties shall jointly review the disagreement. If necessary, settlement shall be reached under the terms of the purchase order.

4.8 RECORDS AND TEST REPORTS

4.8.1 The manufacturer shall maintain a record identifying the raw materials of each lot of coated UC_2 particles.

4.8.2 The system of labelling of coated particle batches and lots and their respective test samples shall provide that:

4.8.2.1 The manufacturer can readily identify each batch and lot with the corresponding samples of particles tested under Section 4.5.1. Records of the Section 4.5.1 tests shall be kept available for two years in case it becomes necessary for the purchaser to examine them.

4.8.2.2 The purchaser can readily relate each lot with the corresponding samples (a) tested under Sections 4.4 and 4.5.2 and (b) supplied to the purchaser for check and referee tests under Section 4.9.1.4.

4.8.3 The manufacturer shall furnish five copies of the certified test report listing in tabular form the results of the tests specified in Sections 4.4.1, 4.4.2, 4.4.3, 4.5.2.1, 4.5.2.2, 4.5.2.3, 4.5.2.6, and 4.5.2.7. The results of all tests specified in this Section 4.8.3 shall be traceable to the lot and sample tested.

[redacted]

4.8.3.1 Results from each density test required (Section 4.4.1) shall be separately reported.

4.8.3.2 Results from the dross counts (Section 4.4.3) shall be reported as the total number of foreign particles per gm.

4.8.3.3 Results from each U analysis required (Section 4.5.2.1) shall be separately reported to the nearest 0.1% U by weight.

4.8.3.4 Results from each carbon analysis required (Section 4.5.2.2) shall be separately reported to the nearest 0.2% by weight.

4.8.4 The manufacturer shall furnish certification of conformance to the requirements of Section 3.4.1 and of Sections 4.6.3.4, 4.6.3.5, 4.6.3.9, and 4.6.3.10. The certifications shall be referenced to the lot and, with the exception of the Section 4.6.3.10 tests, the certifications shall be referenced to the test sample identifications.

4.8.5 Before shipping any lot or container of material not in conformance with this specification, the manufacturer shall obtain from the purchaser a written specification deviation waiving the particular nonconformance of that lot or container of material.

4.9 PREPARATION FOR DELIVERY

4.9.1 Packing

4.9.1.1 Shipping Regulations: Each shipment of coated particles shall be packed to assure (1) the nuclear safety of the shipment and (2) conformance with the pertinent AEC and ICC regulations as referenced in the applicable documents.

4.9.1.2 Method: Before any particles are shipped, the manufacturer's method of packing the coated particles for shipment shall be approved in writing by the purchaser.

4.9.1.3 Lot Segregation: Each package of coated particles shipped shall contain material from one lot only. Lots may not be mixed.

[redacted]

[redacted]

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4.9.1.4 Test Samples: The check test and referee samples for each lot, consisting of the two unused thirds of the 150-gm sample specified in Section 4.6.2.2, shall be packed in two separate containers for submission to the purchaser no later than the date of shipment of the production lot.

4.9.2 Marking: Each shipping container shall be labelled with the PDS Number, including dash number and sub letter; Container, Lot, and Purchaser's Order Numbers; Gross, Tare, and Net Weight; Enrichment; Name of Manufacturer.

[redacted]

APPENDIX

A.1. DETERMINATION OF FINAL PARTICLE SIZE

A.1.1 EQUIPMENT

A.1.1.1 Analytical or Torsion Balance with capacity at least 100 gm and sensitivity 0.01 gm; timer.

A.1.1.2 Tyler Ro-Tap Sieve Shaker with cork knocker; matched Tyler Sieves, 3-inch diameter, stainless steel, in Tyler mesh sizes 60, 65, 100, 170, 200; top cover and bottom pan.

A.1.2 CALIBRATION, CLEANLINESS OF SCREENS

A.1.2.1 Sieves shall be certified to ASTM E-11. Each sieve shall be

(a) visually inspected for possible distortion before every test and
(b) tested monthly with NBS Standard No. 1017 glass spheres.

A.1.2.2 Sieves shall be cleaned before use. Hold sieve to an illuminated screen to check for captured particles. If brushing does not clean satisfactorily, clean in an ultrasonic bath.

A.1.3 PROCEDURE

A.1.3.1 (a) Check and record mesh sizes of sieves as specified in Section A.1.1.2.
(b) Inspect as specified in Section A.1.2.
(c) Assemble sieve stack in order of size, with the coarse sieve on top; set stack in bottom pan and assure fit of top cover.

A.1.3.2 From the manufacturer's 50-gm sample for all of his lot-based tests (Section 4.4.2), take a 10-gm sample and place it in the top sieve and install the top cover.

A.1.3.3 Secure the assembly (Section A.1.3.1 (c) and A.1.3.2) in the shaker and operate the shaker for 15 minutes, timing the cycle with the timer.

A.1.3.4 With the assembly removed from the shaker and set on a horizontal surface, carefully tilt up the top cover and, before removing it, brush its under side to dislodge adhering particles into the top sieve.

A.1.3.5 Similarly, tilt up the top sieve and brush its under side to dislodge adhering particles into the next finer sieve so that they become part of the fraction in the next finer sieve.

A.1.3.6 Weigh the top sieve and contents to the nearest 0.01 gm, and record the value. Subtract the weight of the empty screen from the recorded gross weight. Record the difference as the weight fraction of the particles retained on the screen.

A.1.3.7 Repeat steps A.1.3.5 and A.1.3.6 for each screen below the top one, in downward order to the bottom. Determine the fraction collected in the bottom pan in the manner specified in Section A.1.3.6.

A.1.3.8 The sum of the weights of all fractions (Sections A.1.3.6 and A.1.3.7) must equal not less than 99.0% of the original sample weight, or a rerun must be made with a new sample.

A.1.3.9 The test sheet, showing each fraction weight and its percentage equivalent to the nearest 0.01 w/o (based on the Section A.1.3.8 sum of all fractions), shall be dated and signed by the inspector making the analysis. The data shall show conformance to Section 3.5.2.2, or the lot shall be subject to rejection.

A.2 COATING INTEGRITY DETERMINATION

A.2.1 Method: The determination of acid-soluble uranium in fuel beads using 1-(2-Pyridylazo) 2-naphthol

A.2.2 Apparatus

A.2.2.1 Beckman Model B spectrophotometer with 1-cm cells or equivalent.

A.2.2.2 Beckman Zeromatic pH meter or equivalent.

A.2.2.3 Separatory funnels. (150 ml).

[redacted]

A.2.3 Reagents

A.2.3.1 Disodium Ethylenediaminetetraacetate (EDTA)

A.2.3.2 Potassium Fluoride

A.2.3.3 Aluminum Nitrate Salting Solution:

Dissolve 1800 grams of aluminum nitrate monohydrate in 920 ml of water. When 20 ml of this solution are diluted with 10 ml of demineralized water, the pH should be 0.0 to 0.3. If the diluted solution has a pH outside this range, adjustment is made with concentrated nitric acid or ammonium hydroxide, as required.

A.2.3.4 Tributyl Phosphate 9% Extraction Solution:

A 10 ml portion of Eastman white-label tri-n-butyl phosphate is added to 100 ml of reagent grade chloroform.

A.2.3.5 Pyridine

A.2.3.6 1-(2-Pyridylazo)-2-Naphthol Solution (PAN):

A 0.05% PAN solution is prepared by dissolving 0.050 grams of 1-(2-Pyridylazo)-2-Naphthol (Eastman 7192) in dry methanol. Filter through glass wool and dilute to 100 ml. Store in an amber bottle. The standard curve should be checked each time the reagent is prepared.

A.2.3.7 Standard Uranium Solution - (100 micrograms U/ml):

A portion of N.B.S. U_3O_8 (950A) is placed in a furnace and ignited for one hour at 900°C and desiccated to cool. A standard solution, containing 100 micrograms (ug) of uranium per ml, is prepared by dissolving 0.1181 g of the ignited U_3O_8 in 10 ml of nitric acid and diluting to one liter with water.

A second solution containing 5 ug of uranium per ml is prepared by transferring a 25 ml aliquot of the standard solution (100 ug/ml) to a 500 ml volumetric flask and diluting to volume with water.

A.2.4 Preparation of Standard Curve

Using the two standard solutions, transfer 0.0, 25.0, 50.0, 150.0, 200.0, 250.0, and 300.0 ug of uranium, in 10 ml or less of solution, to 150 ml separatory funnels. Add 0.2 gram of potassium fluoride and 0.2 g of EDTA, swirl to dissolve, and dilute to 10 ml. Add 2 drops of methyl orange and neutralize with 1 to 1 ammonium hydroxide or 1 to 1 nitric acid to the indicator change. Add 20 ml of the aluminum nitrate salting solution and 10 ml of tributyl phosphate extraction solution; shake for two minutes. Filter the organic extract through absorbent cotton into a 25 ml volumetric flask. Repeat the extraction using 5 ml of tributyl phosphate solution, and then wash the aqueous phase with 3 ml of chloroform. Add 5.0 ml of the 0.05% PAN solution and 0.5 ml of pyridine to the combined filtered extracts, and dilute to volume with chloroform. Mix the flasks thoroughly, and after 15 minutes measure the absorbance at 560 mμ using the zero standard as a reference solution. Plot absorbance against micrograms of uranium per 25 ml of solution.

A.2.5 Analysis of Samples

The sample specified in Section 4.5.1 and 4.6.2.1 (or in Section 4.5.2.6, as applicable) is leached for two hours in 50 ml of 50 volume per cent nitric acid and demineralized water at a temperature of 95°C. The acid insoluble portion is removed by filtering through a medium-porosity sintered-glass crucible. The residue is washed with water and the filtrate plus washings are transferred to a 100 ml volumetric flask, and diluted to volume with water. A suitable aliquot is transferred directly into a 150 ml separatory funnel, or, if the aliquot is larger than 10 ml, it is transferred to a 150 ml beaker and evaporated to a volume less than 10 ml and then transferred to the separatory funnel. The sample is then treated as in the preparation of the standard curve, beginning with the addition of 0.2 g of potassium fluoride.

The color is stable for at least 24 hours. A reagent blank must be carried through the entire procedure and used as a reference when the absorbance is measured.

A.2.6 Calculation

A. Per cent acid-soluble uranium of sample weight.

$$\% U = \frac{A}{100 \times B \times C}$$

Where A = micrograms of uranium read from curve.
B = weight of sample in grams.
C = aliquot size in ml.

B. Micrograms of acid-soluble uranium per gram of sample.

$$\frac{\text{microgram uranium}}{\text{gram of sample}} = \%\ U \text{ (from A.)} \times 10^4$$

C. Per cent acid-soluble uranium of original weight of uranium present.

$$\%\ U = \frac{A}{100 \times B \times C \times D}$$

Where A = micrograms of uranium read from curve.
B = weight of sample in grams.
C = Aliquote size in ml.
D = per cent of total uranium present in sample.

A.2.7 Results: The calculated results from item C of Section A.2.6, based on the Section A.2.5 analysis, shall show conformance to Section 3.5.2.5 (a or b, as applicable), or the lot shall be subject to rejection.

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A.3 DETERMINATION OF PARTICLE SHAPE

A.3.1 EQUIPMENT

A.3.1.1 Microradiographic x-ray unit, such as Picker Catalog #905-D.

A.3.1.2 X-ray Camera

A.3.1.3 Microradiographic plates with very high resolution emulsion, such as Kodak High Resolution plates (649-GH).

A.3.1.4 Optical system with camera attachment for making 8 x 10 inch photomicrographs at 150X.

A.3.1.5 Pocket comparator, such as Bausch and Lomb 813435.

A.3.2 CALIBRATION

A.3.2.1 The radiographic setup shall be made such that the geometric unsharpness is less than one micron as computed from the following formula:

$$Ug = \frac{Ft}{d}$$

where:

Ug = geometric unsharpness
F = focal spot size of the X-ray tube in microns
t = object-to-film distance in microns
d = focal-spot-to-object distance in microns

A.3.2.2 The exact magnification of the microscopic lens combination shall be determined by making a photomicrograph of the ruled stage micrometer at the 150X magnification. This photomicrograph shall be used to determine the factor for converting the distance subtended by the pocket comparator reticule to microns.

A.3.3 PROCEDURE

[redacted]
Atomic Energy Act - 1954

A.3.3.1 Preliminary

A.3.3.1.1 From the manufacturer's 50-gm sample for all of his lot-based tests (Section 4.5.2), prepare approximately 50 mg of the particles for a micro-radiographic exposure. Spread the particles as nearly as possible in a one-particle-thick layer in intimate contact with the emulsion side of the Section A.3.1.3 radiographic plate. Make a micro-radiograph of the particles for particle shape measurements.

A.3.3.1.2 Visually scan at least ten fields in the microradiograph at 150X, each field showing at least 50 particles in a monolayer, each particle shown completely within the outer boundaries of the microradiograph.

NOTE: All counts and measurements specified and reported shall be only for particles which are in a monolayer and are completely shown within the outer boundaries of the picture representing them.

A.3.3.1.3 Make photomicrographs at 150X of at least five representative fields, each field showing at least 20 particles as specified in Section A.3.3.1.2. Count and record the number of particles shown in each field, and the total shown in all fields.

A.3.3.2 Measurements: Of the Section A.3.3.1.3 representations, make particle shape measurements with the pocket comparator on all which, as shown, are unsymmetrical (i.e., are not circular, such as to suggest that the actual particles represented are not spherical). For each such particle, measure the major axis as the maximum width of the particle representation and the minor axis as the width perpendicular to the major axis.

A.3.3.3 Computations and Records

A.3.3.3.1 Count and record the number of clumps and sharp-cornered particles shown in the five micro-photographs.

A.3.3.3.2 Compute the major axis to minor axis ratios of all particle representations measured (Section A.3.3.2) but not recorded under Section A.3.3.3.1. Count the number of computations.

A.3.3.3.3 To show conformance to Section 3.5.1, compute and report the following three percentages based on the Section A.3.3.1.3 total: the clumps and sharp-cornered particles recorded under Section A.3.3.3.1; the Section A.3.3.3.2 computations yielding ratios greater than 2; and those yielding ratios greater than 1.5.

A.4 DETERMINATION OF GAMMA SPECTRUM

A.4.1 EQUIPMENT

A suitable gamma ray spectrometer such as:

RIDL - Model 34-12 - 400 channel, or
RIDL - Model 3300 - 100 channel with either tape or electric typewriter readout system or X-Y recorder.
Scintillation crystal such as:
Sodium Iodide, Thallium Activiated, Scintillation Crystal 3" x 3".

A.4.2 PROCEDURE

A.4.2.1 Sample Preparation: From the manufacturer's 50-gm sample for all of his lot-based tests (Section 4.5.2), weigh a 0.5000-gram sample and quantitatively transfer it into a vial. Seal with plastic cap and wipe the outside of the vial clean to prevent contamination of the scintillation crystal.

A.4.2.2 Low Energy Scan: Adjust spectrometer to scan the range from 0 to 230 kev. Place vial on center of scintillation crystal and record spectra for one-minute gross line time. Print out data on either tape or electric typewriter. Plot counts per channel number.

A.4.2.3 High Energy Scan: Adjust spectrometer to scan the range 200 to 1600 kev. Place vial on center of scintillation crystal and record spectra for five-minute gross line time.

Print out data on either tape or electric typewriter. Plot the counts per channel versus channel number. Obtain background scan and plot in a similar manner.

A.4.2.4 Standards: Immediately before making determinations on the Section 4.5.2 material, perform the Section A.4.2.2 and A.4.2.3 scans with 0.5000 gm of NBS 930 standard U_3O_8 in a sealed and cleaned vial as specified in Section A.4.2.1. Report results on standard U_3O_8 along with results on specification material.

A.4.2.5 Acceptance: The results shall show conformance to Section 3.5.2.4, or the lot shall be subject to rejection.

A.5 DETERMINATION OF THERMAL STABILITY

A.5.1 EQUIPMENT

A.5.1.1 A 2300°C furnace with rapid heating and cooling capability.

A 5.1.2 Graphite crucibles, approximately 1/4-inch inside diameter and 1/2-inch in internal height. The crucible shall have a small hole drilled into the external wall or lid to serve as an optical pyrometer target. The hole shall have a depth-to-diameter ratio greater than three.

A.5.1.3 Optical pyrometer (minimum range from 1500°C to 2300°C) such as micro-optical pyrometer Model 95, The Pyrometer Instrument Co., Inc. The resolution of the pyrometer optical t--in shall be sufficient to separate objects with smaller linear dimensions than the diameter of the target in the crucible at the actual pyrometer-to-target distance.

A.5.1.4 Microradiographic X-ray Unit, such as Picker Catalog #805-D.

A.5.1.5 X-ray Camera

A.5.1.6 Microradiographic emulsion, such as Kodak High Resolution plates (649-GH).

[redacted]

A.5.1.7 Optical system with camera attachment for making 8 x 10 inch photomicrographs at 150X.

A.5.1.8 Ruled stage micrometer with line spacing of 0.01 mm.

A.5.1.9 Pocket comparator, such as Bausch and Lomb 813435.

A.5.2 CALIBRATION AND STANDARDIZATION

A.5.2.1 Calibration

A.5.2.1.1 The radiographic setup shall be made such that the geometric unsharpness is less than one micron when calculated by the following formula:

$$Ug = \frac{Ft}{d}$$

Where:

Ug = geometric unsharpness
F = focal spot size of the x-ray tube in microns
t = object-to-film distance in microns
d = focal-spot-to-object distance in microns

A.5.2.1.2 The exact magnification of the microscopic lens combination shall be determined by making a photomicrograph of the ruled stage micrometer at the 150X magnification. This photomicrograph shall be used to determine the factor for converting the distance subtended by the pocket comparator reticule to microns.

A.5.2.1.3 The optical pyrometer shall be calibrated by comparison with a standard pyrometer which has been calibrated by the National Bureau of Standards, the Westinghouse Research Laboratories, or other reference organization approved in writing by the purchaser. The standard pyrometer shall be recalibrated at least yearly.

A.5.2.2 Interlaboratory Standardization: At the option of the purchaser, samples will be furnished to the manufacturer for standardization of the procedure:

A.5.2.2.1 A sample of particles previously thermally treated at 2300°C for four hours, for the manufacturer to perform microradiography and measurement of T, X min., and σ.

A.5.2.2.2 A sample "as coated" for the manufacturer to perform thermal treatment at 2300°C for four hours, microradiography, and measurement of $\bar{X}$, X min., and σ.

A.5.2.2.3 All data obtained in standardization tests performed under this Section A.5.2.2 shall be reported to the purchaser.

A.5.3 PROCEDURE

A.5.3.1 From the manufacturer's 50-gm sample for all of his lot-based tests (Section 4.5.2), obtain a 0.5-to-1.0 gm sample and place it in a crucible as specified in Section A.5.1.2.

A.5.3.2 Put the crucible into the furnace with the axis of the target hole in the line of sight of the pyrometer.* Evacuate or purge the air from furnace and crucible (if purging is used, the method must be approved by the purchaser).

A.5.3.3 Heat to the following cycle in helium or argon maintained at atmospheric pressure: to 2300 ± 25°C in 15 to 20 minutes; hold at that level for four hours ± 10 min.; and cool to 1500°C in 15 to 20 min., and then to room temperature. Determine the 2300 and 1500°C temperatures by sighting the Section A.5.1.3 pyrometer in the target hole of the crucible.*

A.5.3.4 Remove approximately 50 mg of the heat-treated particles (Section A.5.3.3) by riffling, and spread them as nearly as possible in a one-particle-thick layer in intimate contact with the emulsion side of the microradiographic plate. Make a microradiograph of the particles for coating thickness measurements.

* If more than one crucible is loaded into the furnace at a time, provision must be made for sighting into each crucible in turn, or the method for assuring that the Section A.5.3.3 temperature requirement is met in all crucibles shall be submitted to the purchaser for approval, and the approval obtained in writing before the testing is commenced.

[redacted] DATA
[redacted] Act - 1954

A.5.3.5 Visually scan, at 150X, at least ten fields in the microradiograph, each field showing at least 50 particles in a monolayer, each particle shown completely within the outer boundaries of the microradiograph.

NOTE: All counts and measurements specified and reported shall be only for particles which are in a monolayer and are completely shown within the outer boundaries of the picture representing them.

A.5.3.6 Make 8 x 10 inch photomicrographs at 150X of at least eight representative fields, each field showing at least 50 particles as specified in Section A.5.3.5. Count and record the number of particles shown in each field, and the total shown in all fields.

A.5.3.7 Count and record for each field the number of particles in which, as shown, the uranium has diffused completely through the coating at any point. Total and record the sum for the eight fields. To show conformance to the Section 3.5.2.6.2 requirement, compute and record what percentage this sum is of the Section A.5.3.6 total.

A.5.3.8 Number the photomicrographs serially, and orient them by marking "top" and "left" on the appropriate edges. Starting two inches down from the top edge, draw horizontal lines at two-inch intervals to the bottom. Beginning with the top grid line on print number one and scanning the grid lines from left to right, serially record the numbers up to 100 beside those Section A.5.3.5 particle representations which the grid lines intersect.

A.5.3.9 Use the pocket comparator to measure the coating thickness of each of these 100 numbered particles, as follows: Scanning the grid lines from left to right, make the measurement at the point a grid line first intersects each numbered particle representation. Measure radially inward from that point of intersection to determine the thickness of completely unmigrated coating. Compute and record for these 100 thicknesses the average coating thickness ($\bar{X}$); the standard deviation (σ); and (to show conformance to the acceptance criterion of Section 3.5.2.6.1) the value for $\bar{X} - 1.6\sigma$.

[redacted]

A.5.3.10 Use the pocket comparator to make radial measurements of the minimum thickness of unmigrated coating discernible about the circumference of each of the 100 numbered particle representations. Compute and record the average of these minimum thicknesses, X min.

A.5.3.11 Submit copies of all photomicrographs with the sample submitted for each lot. Report the data required to be recorded in Sections A.5.3.6 through A.5.3.10.

[redacted] INFORMATION CATEGORY

[redacted] Authorized Classifier Date

Westinghouse Electric Corporation

Astronuclear Laboratory
P. O. Box 10864
Pittsburgh, Pa. 15236
(Fed. Ident. Code No. 14683)

PURCHASING DEPARTMENT SPECIFICATION 30179-A April 11, 1967
(Not for Publication)

BRAZING POWDER

1. SCOPE

This specification covers the requirements for metal powders for use in brazing, designated as follows:

Designation	Description
PDS-30179-1	Unalloyed molybdenum powder with a mesh size of -325, intended for use in brazing graphite to graphite.

2. APPLICABLE DOCUMENTS

2.1 The following documents, of the issue in effect on the date of invitation for bids, shall form a part of this specification to the extent specified herein:

ASTM B 214

2.2 Copies of ASTM standards may be secured from American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania.

3. REQUIREMENTS

3.1 COMPOSITION

99.50% minimum molybdenum
0.40% maximum oxygen
0.10% maximum all other

3.2 MESH SIZE

-325 mesh (passing 325-mesh sieve) as defined in ASTM B 214.

Printed in U.S.A.
KM/cas

W form 59504A

4. QUALITY ASSURANCE

4.1 SOURCE SURVEILLANCE

4.1.1 Material covered by this specification is subject to source surveillance by a Westinghouse representative. This may include surveillance of the product and of the manufacturer's systems, procedures, and facilities which relate to testing and inspection of this material. The manufacturer shall perform tests and supply data as required to illustrate compliance with all requirements of this specification.

4.1.2 Material accepted by the purchaser's representative at the place of manufacture which is later found to be not in conformance with this specification is subject to rejection.

4.2 COMPLIANCE: No change shall be made in the quality of successive shipments of material furnished under this specification without first obtaining approval of the purchaser.

4.3 CHEMICAL ANALYSIS: Within sixty days prior to shipment of material furnished under this specification, each powder lot shall be analyzed for conformance to Section 3.1.

4.4 TEST REPORTS: The manufacturer shall furnish five copies of a certified test report showing conformance of the material to Section 3. The report shall show the Purchase Order Number; P.D. Specification Number, including dash number; Lot or Test Identification Number; Date of Test; Name of Manufacturer.

5. PREPARATION FOR DELIVERY

Each shipping container shall be plainly marked with the Purchase Order Number; P.D. Specification Number; Lot Number; Gross, Tare and Net Weight; Name of Manufacturer.

6. PURCHASER STORAGE

The purchaser will affix a tag to each container of material, the tag bearing the date of receipt and instructions prohibiting use of the material later than one year from the date of receipt unless a purchaser analysis, performed within a year prior to such use, shows conformance to Section 3.1.

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INFORMATION CA[redacted]ORY

[redacted] -RD

[redacted] 2/22/68

Authorized Classifier [redacted]

Westinghouse Electric Corporation

Astronuclear Laboratory
P. O. Box 10864
Pittsburgh, Pa. 15236
(Fed. Ident. Code No. 14683)

PURCHASING DEPARTMENT SPECIFICATION 30106-E

June 24, 1968

(Not for Publication)

NIOBIUM CARBIDE/GRAPHITE COMPOSITE MATERIAL - HOT PRESSED

(Title Unclassified)

1. SCOPE

(C) This specification covers requirements for hot pressed niobium carbide-graphite composite material, designated as follows:

Designation	Description
30106-1	Hot pressed niobium carbide-graphite composite material (Nominal Composition 75 w/o NbC and 25% w/o graphite)
30106-2	Hot pressed niobium carbide-graphite low expansion material (Nominal Composition 75 w/o NbC and 25% w/o Carbon) pressed for 90 minutes.
30106-3	Hot pressed niobium carbide-graphite low expansion material (Nominal Composition 75 w/o NbC and 25% w/o Carbon) pressed for 15 minutes with a modified heat treating cycle.

NOTE: Unless otherwise specified, the requirements of this specification apply to all designations.

2. APPLICABLE DOCUMENTS

(U) The following documents, of the issue in effect on the date of invitation for bids, shall form a part of this specification to the extent specified herein.

PDS-30104 PDS-30046 PS-294514 ASTM B214-64
PS 597951

3. REQUIREMENTS

3.1 MANUFACTURE

3.1.1 Materials:

(U) 3.1.1.1 The niobium carbide powder shall conform to PDS-30104-3.

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(C) 3.1.1.2 Graphite for 30106-1

The graphite flour shall conform to PDS-30046-1 except that the size fraction shall be between 150 and 325 mesh. A representative sample of this screened flour shall be particle size analyzed in accordance with ASTM B214-64. This analysis shall reveal not more than 8% through a 325 mesh screen and not more than 2% retained on a 150 mesh screen.

Carbon for 30106-2 and -3

(U) a) The form of carbon shall be calcined petroleum coke (needle coke) having a high degree of crystallinity and an elongated particle shape.

(U) b) The carbon shall not contain impurities in excess of the following limits.

Impurities	Max. Value
Total Ash	0.15 percent (w/o)
Sulfur	1.20 percent (w/o)
Si	200 ppm
Fe	200 ppm
V	40 ppm
Ti	40 ppm
Al	100 ppm
Mn	40 ppm
Ni	40 ppm
Ca	40 ppm
Mg	40 ppm
Co	100 ppm
Cr	40 ppm

(C) c) The particle size when analyzed in accordance with ASTM-B214-64 shall conform to the following requirements:

Screen Size	Percent Through, By Weight
-100	100
-150	50
-325	less than 8

d) A one pound sample shall be furnished to the purchaser for approval prior to producing billets.

3.1.2 Mixing:

(U) 3.1.2.1 The NbC powder and graphite flour for 30106-1 billets or carbon for 30106-2 or -3 billets shall be thoroughly mixed to assure an even distribution of the niobium carbide and graphite particles.

(U) 3.1.2.2 The mixture shall not be subjected to any practices which could result in segregation prior to hot pressing.

(U) 3.1.2.3 If wet blending is used, no material shall be added that will result in a residue after hot pressing. In addition, the procedure and vehicle used shall be approved by the purchaser.

3.1.3 Hot Pressing:

(C) 3.1.3.1 The powder mixture shall be charged into a cylindrical graphite die and pressed axially with double-acting graphite plungers in an Argon atmosphere under the following conditions:

	30106-1	30106-2	30106-3
Temperature (at billet centroid)	3100 + 50-75°C	3100 + 50-75°C	3100 + 50-75°C
Pressing Pressure on compact	2800 psi min.	2800 psi min.	2800 psi min.
Heat up time	4 hours max.	4 hours max.	4 hours max.
Time (at temperature and stress	20 minutes min.	90 minutes ± 1 min.	15 minutes + 1 min.

(U) 3.1.3.2 The pressing temperature shall be the true temperature of the composite material; all necessary corrections shall be applied to the optical measurement including sight glass, emmittance and instrument errors. In addition, the temperature measurement equipment shall have been calibrated using the zirconium carbide-graphite eutectic melting temperature, 2890°C. Equal parts by weight of zirconium carbide and graphite powder shall be mixed and two pressings made, one at a temperature of 2840°C and one at 2940°C. Pressing pressure and time shall be as specified in Section 3.1.3.1. The pressings shall be examined microstructurally for melting. A sample representative of one-half of each pressing, in the form of a longitudinal section, shall be submitted to the purchaser for approval of the calibration prior to the start of a series of production runs.

(U) 3.1.3.3 Neither the axial nor the diametral temperature gradient shall exceed 150°C as revealed by the temperature calibration of Section 3.1.3.2.

(C) 3.1.3.4 The pressing shall not be removed from the die until a temperature of 600°C, or less, is reached. Cooling to 600°C shall be by radiation only.

(C) 3.1.4 Heat Treatment: (30106-1 and -2)

Each billet shall be given a post-pressing heat treatment in a stress-free condition for one hour minimum at a temperature of 2500°C ± 50° in an argon environment.

(30106-3)

Each billet shall be given a post-pressing heat treatment under a compressive stress of 2000 psi for one hour minimum at temperature of 2700°C ± 50° in an argon atmosphere. The billet shall not be surrounded by a die body or any structure that would impede lateral or axial movement.

(U) 3.1.5 Dimensions: The dimensions of the billet shall be in accordance with the applicable drawing.

(U) 3.1.6 Identification: The top of the billet as pressed shall be identified by the purchaser's assigned serial number with a vibrating tool or other approved permanent type of identification.

(C) 3.2 COMPOSITION: The chemical composition shall be within the range of 74 to 76 weight percent niobium carbide. The metallic impurities shall not exceed the limits specified in PDS-30104-3.

3.3 DENSITY

(C) 3.3.1 The bulk density of each billet shall be not less than 4.40 gms/cc (See Section 4.3.3)

(U) 3.3.2 Density determinations shall be procured for engineering information on the chemistry samples and on all flexural and creep test specimens. This information shall form a part of the reports on the results of the chemical analysis, flexural and creep tests.

(C) 3.4 FLEXURAL STRENGTH: The average minimum room temperature with-grain and across-grain flexural strength of each billet shall be 10,000 and 6,700 psi respectively. (See Section 4.3.4)

(C) 3.5 CREEP STRENGTH: The across-grain compressive creep deformation shall be 8% maximum (See Section 4.3.5).

(C) 3.6 THERMAL EXPANSION

30106-1 Expansion

The total with-grain thermal expansion shall be not more than 2% over the temperature range of 25°C to 2500°C (See Section 4.3.6).

30106-2 & -3 Expansion

The total with-grain thermal expansion shall be not more than 1.8% over the temperature range of 25°C to 2500°C (See Section 4.3.6).

(C) 3.7 HOMOGENEITY: The billet shall not contain more than four high density inclusions with a length greater than 0.060 inches in any square inch as revealed by a 10X microstructural examination or by a radiographic examination. (See Section 4.3.7 and 4.3.8).

4. QUALITY ASSURANCE

4.1 SURVEILLANCE

(U) 4.1.1 Material covered by this specification is subject to source surveillance by a Westinghouse representative. This may include surveillance of the product and of the manufacturer's systems, procedures and facilities which relate to testing and inspection of this material. The manufacturer shall perform tests and supply data as required to illustrate compliance with all requirements of this specification.

(U) 4.1.2 Material accepted by the purchaser's representative at the place of manufacture which is later found to be not in conformance with this specification is subject to rejection.

(U) 4.2 COMPLIANCE: No change shall be made from this specification without first obtaining the purchaser's approval.

4.3 TESTS AND INSPECTION

(U) 4.3.1 Specimens: All specimen locations and dimensions shall be in accordance with the applicable drawing. All specimens shall be identified so as to indicate the billet number and location within the billet.

(U) 4.3.2 Chemical Analysis: Chemical analysis shall be determined on a sample procured from the top and bottom of every tenth billet. The analysis shall be determined by a method approved by the purchaser.

(U) 4.3.3 Density: The density shall be determined by weighing and measuring or by immesion.

4.3.4 Flexural Strength:

(U) 4.3.4.1 The flexural strength shall be determined on a minimum of three with-grain specimens from each billet. In addition, duplicate across-grain measurements shall be made at the top, middle and bottom of each billet.

(U) 4.3.4.2 Four point loading shall be used. The spacing of the load points shall form a part of the records.

(U) 4.3.4.3 The test technique shall be approved by the purchaser.

4.3.5 Creep Testing:

(U) 4.3.5.1 The creep strength shall be determined on a minimum of one specimen from the top and bottom of each billet.

(U) 4.3.5.2 Specimens shall be cylindrical with a length to diameter ratio of two to one.

(U) 4.3.5.3 The percentage $\Delta L/L$ deformation shall be determined under an axial compressive stress of 4000 psi at 2500°C ± 50°C for one hour. The test technique shall be approved by the purchaser.

(U) 4.3.6 Thermal Expansion Testing:

4.3.6.1 The thermal expansion shall be determined on one specimen from the first and fifth billet and every tenth billet thereafter from each billet lot.

4.3.6.2 Linear growth during the test shall be measured using an optical technique.

4.3.6.3 The test specimen dimension and test technique shall be approved by the purchaser.

(U) 4.3.7 Radiography: A radiograph shall be taken of each billet, in a direction perpendicular to the pressing direction, in accordance with PS-294514 at the two percent sensitivity level. In addition, a radiograph shall be taken of each flexural and creep test specimen, for information only, on the first five billets and every tenth billet thereafter. Radiographic standards shall be as specified in PS-597951.

(U) 4.3.8 Microstructure: A sample disc shall be secured from the first five billets for initial qualification. After purchaser's approval of initial microstructures, sampling shall be on the basis of every tenth billet. A 10X photograph of the worst area shall be submitted to the purchaser. Microstructural standards shall be as specified in PS-597951.

(U) 4.4 SAMPLES: After the test specimens have been procured from the sample disc, the remainder shall be properly identified and forwarded to the purchaser.

(U) 4.5 TEST REPORTS: The manufacturer and/or evaluator shall furnish five copies of a certified test report showing the results of the tests specified and bearing a statement that the material conforms to this specification. The report shall show the Purchase Order Number; PDS-30106 (including the sub-letter and dash number); Dimensions; Lot Identification Numbers; Date of Manufacture and Test; Name of Manufacturer.

(U) 5. PREPARATION FOR DELIVERY

5.1 MARKING: Shipping containers shall be plainly marked as follows: Purchase Order Number; PDS-30106 (with applicable dash number); Dimensions; Lot Identification Numbers; Gross, Tare, and Net Weight; Name of Manufacturer.

5.2 PACKING: The billets shall be packed in appropriate containers to prevent contamination and physical damage during shipment.

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Authorized Classifier Date

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Westinghouse Electric Corporation

Astronuclear Laboratory
P. O. Box 10864
Pittsburgh, Pa. 15236
(Fed. Ident. Code No. 14683)

PURCHASING DEPARTMENT SPECIFICATION 30104-E
(Not for Publication)

February 9, 1968

NIOBIUM CARBIDE POWDER
(Title Unclassified)

(U) 1. SCOPE

This specification covers requirements for niobium carbide powder, designated as follows:

PD Spec Designation	Description
30104-1	Cancelled - replaced by 30104-3
30104-2	Cancelled - replaced by 30104-4
30104-3	Niobium carbide powder for hot pressed composite material.
30104-4	Niobium carbide powder for the matrix of fuel elements.

NOTE: Unless otherwise specified, the following requirements apply to all designations.

(C) 2. APPLICABLE DOCUMENTS

2.1 The following documents, of the issue in effect on the date of invitation for bids, shall form a part of this specification to the extent specified herein:

ASTM B-214 ASTM B-215 ASTM F6
ASTM B-329 ASTM B-330

2.2 Copies of ASTM standards may be secured from American Society for Testing and Materials, 1916 Race Street, Philadelphia, Pennsylvania.

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3. REQUIREMENTS

(U) 3.1 GENERAL

3.1.1 Condition: The powder shall be free from foreign material and contamination. Suitable precautions shall be taken to prevent pickup of such contamination during processing.

3.1.2 Lubricants: Lubricants shall not be used in the processing of this powder.

(C) 3.2 CHEMICAL COMPOSITION: The powder shall conform to the following composition:

30104-3 Analysis

Constituent	Weight, Per Cent Min.	Max.
NbC	99.00	
Combined Carbon	10.8	11.6
Free Carbon		0.40
Tantalum		0.50
Tungsten		0.10
Other Metallic Impurities, Each		0.03
Other Metallic Impurities, Total		0.10

30104-4 Analysis

Constituent	Weight, Per Cent Min.	Max.
NbC	99.40	
Combined Carbon	10.8	11.6
Tantalum		0.10
Silicon		0.005
Iron		0.005
Cobalt		0.005
Boron		0.0003
Other Metallic Impurities, each		0.10
Other Metallic Impurities, total		0.30

(C) 3.3 DENSITY

3.3.1 Apparent Density: The apparent density shall be 2.7 g/cc minimum.

3.3.2 Tap Density:
(30104-3) The tap density shall be 4.00 g/cc minimum.
(30104-4) The tap density shall be 3.5 g/cc minimum.

(C) 3.4 SIEVE SIZE: Each powder lot shall be prepared from powder that conforms to the following sieve analysis:

Mesh Size	% by Weight
-325	99.0 min.
+270	1.0 max.
+100	0.0 max.

(C) 3.5 PARTICLE SIZE: The average particle size (Fisher Sub Sieve Size) shall be within the following range:

30104-3	4.0 to 7.0 microns
30104-4	3.0 to 5.5 microns

(U) 3.6 POROSITY: The microstructure of a sample from each powder lot, examined at 400X magnification, shall not reveal porosity in excess of 10 percent within the powder particles.

4. QUALITY ASSURANCE

(U) 4.1 SOURCE SURVEILLANCE

4.1.1 Material covered by this specification is subject to source surveillance by a Westinghouse representative. This may include surveillance of the product and of the manufacturer's systems, procedures, and facilities which relate to testing and inspection of this material. The manufacturer shall perform tests and supply data as required to illustrate compliance with all requirements of this specification.

4.1.2 Material accepted by the purchaser's representative at the place of manufacture which is later found to be not in conformance with this specification is subject to rejection.

(U) 4.2 COMPLIANCE: No change shall be made in the quality of successive shipments of material furnished under this specification without first obtaining approval of the purchaser.

(U) 4.3 SAMPLING

4.3.1 The preparation of powder lots for sampling shall be in accordance with ASTM B-215. A lot shall consist of a batch of powder processed together by the manufacturer's standard practice, identified by a batch or test identification number, and presented for inspection at one time.

4.3.2 Each powder lot shall be subject to evaluation by the purchaser for conformance to the specification as a final acceptance criteria. A 5 pound sample shall be taken from the lot, using an approved sampling procedure, and submitted to the purchaser.

(U) 4.4 CHEMICAL ANALYSIS

4.4.1 Each powder lot shall be analyzed and conform to the chemical composition limits specified in Section 3.2.

4.4.2 Chemical analysis shall be determined in accordance with ASTM Standards or by a method approved by the purchaser. The presence of iron and other trace elements normally associated with the manufacture of this powder shall be determined by spectroscopic analysis.

(U) 4.5 APPARENT AND TAP DENSITY

4.5.1 The apparent density of each powder lot shall conform to the requirements of Section 3.3.1 when tested in accordance with ASTM B-329.

4.5.2 The tap density of each powder lot shall conform to the requirements of Section 3.3.2 when tested in accordance with ASTM F6.

(U) 4.6 SIEVE ANALYSIS: The sieve size of each powder lot shall conform to Section 3.4 when tested in accordance with ASTM B-214.

(U) 4.7 PARTICLE SIZE: The particle size of each powder lot shall conform to Section 3.5 when tested in accordance with ASTM B-330.

(U) 4.8 MICROSTRUCTURE: A photomicrograph shall be taken of the mounted and polished sample examined in Section 3.6 for submission to the purchaser.

(U) 4.9 TEST REPORTS: The manufacturer shall furnish five copies of a test report showing the results of the tests specified in Sections 3.2 to 3.5 inclusive, and bearing a statement that the powder conforms to this specification. The report shall also include the photomicrograph of Section 4.8. The report shall show the Purchase Order Number; PD Specification Number, including dash number; Lot or Test Identification Number; Date of Test; Name of Manufacturer.

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Atomic Energy Act - 1954

5. PACKING AND MARKING

(U) 5.1 PACKING: The powder shall be packed in sealed plastic containers to prevent contamination during shipment and storage. The plastic containers shall be loaded into metal containers for shipment.

(U) 5.2 MARKING: Each shipping package shall be plainly marked with the Purchase Order Number; P D Specification Number; Lot Number; Gross, Tare, and Net Weight; Name of Manufacturer.

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Atomic Energy Act - 1954

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(N for Publication)

Westinghouse Electric Corporation

Astronuclear Laboratory

P. O. Box 10864

Pittsburgh, Pa. 15236

(Fed. Ident. Code No. 14683)

June 22, 1966

COLUMBIUM PENTACHLORIDE

1. SCOPE

This specification covers the requirements for columbium pentachloride, designated as follows:

P D Spec Designation	Description
30052-1	Columbium pentachloride powder or granules

2. APPLICABLE DOCUMENTS: None

3. REQUIREMENTS

3.1 CHEMICAL COMPOSITION: The material shall be at least 99.0% columbium pentachloride, and the content of the impurities listed in the following tabulation shall be less than the limits indicated, based on columbium by weight.

Impurity	Limit, ppm
Boron	1
Cobalt	25
Iron	50
Silicon	100
Vanadium	100
Titanium	300
Zirconium	300
Tantalum	500
Tungsten	500

3.2 MAXIMUM PARTICLE SIZE: The material shall be fine enough to pass through a 2 1/2 mesh Tyler sieve.

4. QUALITY ASSURANCE

4.1 SOURCE SURVEILLANCE

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4.1.1 Material covered by this specification is subject to source surveillance by a quality representative of the purchaser. This will include surveillance of the product and of the seller's systems, procedures, and facilities which relate to the manufacture and inspection of this material. The seller shall perform tests and supply data, at no extra cost, as required to illustrate compliance with all requirements of this specification.

4.1.2 Material accepted by the purchaser's inspector at the place of manufacture which is later found to contain imperfections not detected at the place of manufacture, or which subsequent tests or analysis show to be not in accordance with this specification, is subject to rejection.

4.2 CONSISTENT QUALITY: No change shall be made in the quality of successive shipments of material furnished under this specification without first obtaining the approval of the purchaser.

4.3 APPEARANCE: The material shall be uniformly bright yellow in color; no oxychloride formation or extraneous materials shall be visually evident.

4.4 CHECK ANALYSIS: Each lot of material furnished under this specification is subject to a check analysis by the purchaser to assure conformance with Section 3.1.

4.5 SAMPLING PLAN

4.5.1 A sample shall be taken by continuously accumulating from the process stream at least 300 grams of material from each lot, such that the sample obtained is representative of the entire lot.

4.5.2 At least 200 grams of the sample shall be inspected by the supplier to establish conformance with the requirements of Sections 3.2 and 4.3.

4.5.3 At least 100 grams of the sample shall be provided by the supplier for purposes of check analysis under Section 4.4.

4.5.4 A lot shall consist of all the material continuously processed together and inspected at one time.

4.6 REJECTION STANDARDS: Any lot of material found not to be in conformance with the requirements of Sections 3 and 4.3, when inspected in accordance with Sections 4.4 and 4.5, is subject to rejection.

4.7 REPORTS: The manufacturer shall furnish five copies of a certified report showing the results of the inspection specified in Section 4.5.2, and bearing a statement that the material conforms to the requirements of Section 3.1 and this specification. The report shall show the Purchase Order Number; PDS 30052 (including the sub-letter and dash number); Inspection Identification Number; Date of Inspection; Name of Manufacturer.

5. PREPARATION FOR DELIVERY

5.1 MARKING: Each container shall be plainly marked with the Purchase Order Number; PDS 30052 (including the sub-letter and dash number); Lot Number; Container Number _____of _____ (Number of) containers of each lot in each shipment; Container Number containing the sample required by Section 4.5.3; Gross, Tare, and Net Weight; Name of Manufacturer

5.2 PACKING

5.2.1 All material shall be loaded, stored, and shipped under a positive pressure of argon or nitrogen. Shipping containers shall be pressurized to a minimum of 3 psig argon or nitrogen.

5.2.2 The sample required in Section 4.5.3 shall be separately packaged, sealed in suitable container, and placed in the head of one of the shipping containers containing the lot of material represented. The container in which the sample has been included shall be clearly marked on the head of the container to indicate the presence of the sample.

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INFORMATION CATEGORY

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Authorized Classifier Date

Westinghouse Electric Corporation

Astronuclear Laboratory
P. O. Box 10864
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(Fed. Ident. Code No. 14683)

PURCHASING DEPARTMENT SPECIFICATION 30131
(Not for Publication)

6/17/68

RESEARCH COATING COMPOUND
(Title Unclassified)

(C) 1. SCOPE

This specification covers the requirements for resublimed zirconium tetrachloride, designated as follows:

PD Spec Designation	Description
30131-1	Zirconium tetrachloride powder

(U) 2. APPLICABLE DOCUMENTS - None

3. REQUIREMENTS

(C) 3.1 CHEMICAL COMPOSITION: The powder shall be at least 99.8 percent by weight of zirconium tetrachloride. The maximum impurity content shall be as follows:

Impurity	Limit, ppm
Boron	2
Cadmium	0.5
Cobalt	5
Chromium	100
Copper	25
Iron	50
Hafnium	70
Manganese	15
Molybdenum	60
Nickel	90
Oxygen	2000
Silicon	300
Tantalum	200
Titanium	30
Vanadium	20
Tungsten	25

(C) 3.2 PARTICLE SIZE: The particle size of each powder lot shall be fine enough that a minimum of 95%, by weight, passes through a 100 mesh Tyler sieve.

4. QUALITY ASSURANCE

(U) 4.1 SOURCE SURVEILLANCE

4.1.1 Material covered by this specification is subject to source surveillance by a Westinghouse representative. This may include surveillance of the product and of the manufacturer's systems, procedures, and facilities which relate to testing and inspection of this material. The manufacturer shall perform tests and supply data as required to illustrate compliance with all requirements of this specification.

4.1.2 Material accepted by the purchaser's representative at the place of manufacture which is later found to be not in conformance with this specification is subject to rejection.

(U) 4.2 COMPLIANCE: No change shall be made in the quality of successive shipments of material furnished under this specification without first obtaining approval of the purchaser.

(U) 4.3 SAMPLING:

4.3.1 A sample shall be taken by continuously accumulating from the process stream at least 300 grams of material from each lot, such that the sample obtained is representative of the entire lot.

4.3.2 At least 200 grams of the sample shall be inspected by the supplier to establish conformance with the requirements of Section 3.

4.3.3 At least 100 grams of the sample shall be provided by the supplier for inspection by the purchaser (see Section 5.2.4).

4.3.4 A lot shall consist of the contents of each 53 gallon container shipped.

(U) 4.4 REJECTION: Each lot of material furnished to this specification shall be subject to check analysis and a particle size determination by the purchaser. A lot of material found to be not in conformance with the requirements of Section 3 and 5.2.1 shall be subject to rejection.

(U) 4.5 TEST REPORTS: The manufacturer shall furnish five copies of a certified report showing the results of the inspection specified in Sections 4.3.2 and 5.2.2 and bearing a statement that the material conforms to the requirements of Section 3 of this specification. The report shall show the Purchase Order number; Inspection Identification Number; PDS 30131 (including sub-letter and dash number); Date of Inspection; and Name of Manufacturer.

5. PREPARATION FOR DELIVERY

(U) 5.1 MARKING: Each container shall be plainly marked with the Purchase Order Number; Gross, Tare, and Net Weight; Name of Manufacturer.

(U) 5.2 PACKING

5.2.1 All material shall be loaded, stored, and shipped under a positive pressure of argon. Shipping containers shall be pressurized in the range of 3 to 6 psig.

5.2.2 The pressure (3 to 6 psig) specified under Section 5.2.1 shall be checked by the supplier through the valve assembly specified under Section 5.2.5.2 as follows:

5.2.2.1 At the time of packaging each container.

5.2.2.2 At the time of shipment of each container.

5.2.3 No container shall be shipped whose pressure has dropped below 3 psig.

5.2.4 The sample required in Section 4.3.3 shall be separately packaged, sealed in a suitable container, and placed in the head of the shipping container specified in Section 5.2.5.

5.2.5 The material shall be packaged and sealed in 53 gallon metal containers. Each of these containers shall be equipped as follows:

5.2.5.1 A discharge valve for the container contents shall be provided which is readily connectable to a 2-inch pipe nipple.

5.2.5.2 A valve assembly for checking container pressure shall be provided which consists of the following items:

5.2.5.2.1 3/4 NPT male to 1/4 NPT female reducer, Type 304 Stainless Steel.

5.2.5.2.2 Hoke valve, Catalog Number 30201-1.

5.2.5.2.3 1/4 NPT closed nipple, Type 304 Stainless Steel.

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OF 1954

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Astronuclear Laboratory
P. O. Box 10864
Pittsburgh, Pa. 15236
(Fed. Ident. Code No. 14683)

MP-1

PURCHASING DEPARTMENT SPECIFICATION 30095
(Not for Publication)

March 24, 1966

POWDER, ALLOY - COATING

1. SCOPE

This specification covers molybdenum hexacarbonyl powder designated as follows:

PD Spec. Designation	Description
30095-1	Powder containing 98.0% minimum molybdenum hexacarbonyl.

2. APPLICABLE DOCUMENTS

2.1 The following documents, of the issue in effect on the date of invitation for bids, shall form a part of this specification to the extent specified herein.

ASTM A214

2.2 Copies of ASTM Standards should be obtained from the American Society for Testing and Materials, 1916 Race Street, Philadelphia 3, Pennsylvania.

3. REQUIREMENTS

3.1 MANUFACTURE

3.1.1 The material shall be steam distilled in portions of about 100 pounds each.

3.1.2 When specified on the purchase order, the manufacturer shall supply a 10-gram sample from each steam distilled portion for informational testing.

3.1.3 After steam distillation, the material shall be dried in batches of 5 to 10 pounds each.

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3.1.4 After the material is dried, it shall be sampled as specified in Section 4.3.

3.2 CHEMICAL COMPOSITION: The material shall conform to the following composition:

Constituent	Limit (based on total weight of material)
Molybdenum hexacarbonyl	98.00% min.
Boron	*
Beryllium	*
Cadmium	*
Chromium	*
Copper	*
Iron	*
Nickel	*
Niobium	*
Lead	*
Silicon	*
Thorium	*
Tin	*
Tungsten	*
Vanadium	*
Zinc	*
Zirconium	*
Total metallic impurities	1000 ppm max.
Insoluble residue **	0.10% max.

* Analyze and report for information only

** As determined according to Section 4.4.2

3.3 PHYSICAL PROPERTIES

3.3.1 Particle Size: The particle size shall be determined for information only as specified in Section 4.5.1.

3.3.2 Vapor Pressure: The vapor pressure at 100°C and at 150°C shall be determined for information only according to Section 4.5.2.

3.3.3 Bulk Density: The bulk density at room temperature shall be determined for information only according to Section 4.5.3.

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4. QUALITY ASSURANCE

4.1 SOURCE SURVEILLANCE

4.1.1 Material covered by this specification is subject to source surveillance by a Westinghouse quality representative. This may include surveillance of the product and of the manufacturer's systems, procedures, and facilities which relate to the manufacture and inspection of this material.

4.1.2 Material which tests or analysis by the purchaser show to be not in conformance with this specification, is subject to rejection.

4.2 CONSISTENT QUALITY: No changes shall be made in the quality of successive shipments of material furnished under this specification without first obtaining the approval of the purchaser.

4.3 SAMPLING

4.3.1 From each batch of dried material, the manufacturer shall take a sample consisting of about 10% of the 5 to 10 pound product.

4.3.2 The samples from all of the batches making up a lot shall be combined to form a composite sample weighing five to ten pounds.

4.3.3 A lot shall consist of all the product with the same processing history, originating from the same raw materials, and shipped to the purchaser in a single container.

4.3.4 The manufacturer, after blending the composite sample for at least 30 minutes in a rotary blender, shall ship about 250 grams of it to the purchaser for acceptance analysis and testing.

4.3.5 After obtaining sample material for his analysis and testing from the composite sample, the manufacturer shall add the remainder of the composite sample to the lot to be shipped.

4.3.6 Portions of the composite sample shall be used for the analyses and tests specified in Sections 4.4 and 4.5.

4.4 CHEMICAL PROPERTIES

4.4.1 Chemical Analysis: The amount of the constituents specified in Section 3.2 other than molybdenum hexacarbonyl and insoluble residue shall be determined by wet chemical or spectrographic methods.

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4.4.2 Insoluble Residue: A portion of the composite sample shall be dissolved in chloroform or other suitable solvent. The insoluble residue shall be determined by centrifuging or filtering the solution and weighing the residue.

4.4.3 Examination by X-Ray Diffraction: Examination by X-ray diffraction shall be used to verify that the basic material is molybdenum hexacarbonyl.

4.4.4 Examination for Other Carbonyls: Examination by infra-red analysis shall be used to detect carbonyls other than molybdenum hexacarbonyl and to determine their amount if measurement is practicable, for information only.

4.5 DETERMINATION OF PHYSICAL PROPERTIES

4.5.1 Particle Size: The distribution of particle sizes shall be determined in accordance with ASTM B214, using a 325-mesh sieve as that with the smallest openings.

4.5.2 Vapor Pressure: Determination shall be according to a method submitted by the manufacturer for the purchaser's approval.

4.5.3 Bulk Density: Determination shall be according to a method submitted by the manufacturer for the purchaser's approval.

4.6 REJECTION: A lot represented by a sample not conforming to a requirement of Section 3.2, when analyzed according to Section 4.4.1, 4.4.2, or 4.4.3, shall be subject to rejection.

4.7 RECORDS: The manufacturer shall maintain complete records of the processing steps and retain them for two years. They shall be made available to the purchaser upon request.

4.8 CERTIFICATION: The manufacturer shall furnish five copies (one copy, attention Materials Department) of a certification that the sample, as shipped to the purchaser according to Section 4.3.4, was prepared as specified in Sections 4.3.1, 4.3.2 and 4.3.4 and is representative of the lot of material. The report shall show the Purchase Order Number; P D Specification Number, including dash number; Lot Number; Name of Manufacturer.

5. PREPARATION FOR SHIPMENT

5.1 PACKING AND SHIPPING

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5.1.1 Sample Material: The sample designated in Section 4.3.4 shall be packed in a sealed inner container which excludes moisture and contamination and a rigid outer container which protects the inner container from physical damage during shipment.

5.1.2 Production Material: Each lot of production material shall be placed in a sealed steel container with a polyethylene liner fused to the inner surface, or in a sealed rigid polyethylene chamber, fitted into a steel container. The material shall occupy no more than two thirds of the volume of the container. The container shall be sufficiently rugged to withstand rototumbling for blending without breaking the seal or distorting the container. The container shall be crated or otherwise protected from damage during shipment.

5.2 MARKING: Each outer container shall be plainly marked with the Purchase Order Number; P D Specification Number; Lot Number; Gross, Tare, and Net Weight; Name of Manufacturer.

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Westinghouse Electric Corporation

(File 52217AP) Nov 5, 1964

Purchasing Department Specification 52217AP (Sub B) (Federal Code 79500)

ANHYDROUS HYDROGEN CHLORIDE

1. This specification covers anhydrous hydrogen chloride gas with a minimum purity of 99.5 per cent.

2. No change shall be made in the quality of successive shipments of material furnished under this specification without first obtaining the approval of the purchaser.

CHEMICAL PROPERTIES AND TESTS

3. The material shall conform to the following purity requirements:

Hydrogen Chloride, per cent by vol, min	99.5
Carbon Dioxide, per cent by [illegible] max	0.030
Oxygen, per cent by vol, max	0.042
Organics (by I.R.), per cent by wt, max	0.0010
Free Chlorine, per cent by vol, max	0.0001
Moisture, per cent by wt, max	0.0200

4. CHECK ANALYSIS. Analyses may be made by the purchaser on each shipment of material. The composition thus determined shall conform to the requirements specified in Section 3. Material not conforming to the requirements shall be subject to rejection.

CYLINDERS

5. CYLINDERS. (5.1) The material shall be supplied in cylinders that meet the requirements of the Bureau of Explosives and Rulings of I.C.C.
(5.2) The hydrogen chloride shall be supplied at a cylinder pressure equal to its own vapor pressure, 613 psig at 70 F (21.1 C).

6. OUTLET VALVE CONNECTION. Unless otherwise agreed upon between the purchaser and the manufacturer the outlet valve connection shall conform to ASA 57.1 CGA Connection 330.

MARKING

7. MARKING. (7.1) Each cylinder shall be marked in accordance with ASA Z48.1.
(7.2) Each cylinder shall bear a tag plainly marked as follows. Purchase Order Number; Anhydrous Hydrogen Chloride, P D Spec 52217AP (Sub B); Volume; Name of Manufacturer.

Westinghouse

ELECTRIC CORPORATION

Purchasing Department Specification 52118BA (Sub A) April 5, 1960
(Not for publication)

HIGH PURITY LIQUID ARGON

1. This specification covers high purity liquid argon in a special, insulated cylinder.

2. No change shall be made in the quality of successive shipments of material furnished under this specification without first obtaining the approval of the purchaser.

CHEMICAL PROPERTIES AND TESTS

3. The gas mixture which results when the liquid argon is evaporated shall conform to the following requirements:

Argon, Min	99.995 Per cent
Hydrogen, Max	5 PPM
Oxygen, Max	5 "
Nitrogen, Max	10 "
H_2O, Max	1 "
Carbon bearing gases, Max	5 "

4. CHECK ANALYSIS: Analyses may be made by the purchaser on each lot. The composition thus determined shall conform to the requirements specified in Section 3. Lots not conforming to those requirements shall be subject to rejection.

CYLINDERS

5. CYLINDERS: (5.1) The cylinders shall meet the requirements of the Bureau of Explosives and Rulings of I.C.C.
(5.2) The cylinders shall have a minimum capacity that is equal to the equivalent of 2500 std. cu ft of gaseous argon.
(5.3) (5.3.1) The cylinders shall be insulated sufficiently to limit the mean rate of internal pressure increase during periods of non-use to 3 psig per hour.
(5.3.2) To prevent undue pressure build-up due to evaporation during periods of non-use, a relief valve shall be provided to bleed off pressure after a minimum period of 60 hours, or at a maximum pressure of 250 psig. The valve shall automatically reclose to prevent contamination of the remainder of the contents.
(5.4) The cylinders shall be adequately protected against rupture due to internal pressure build-up and failure of the relief valve specified in Section 5.3.2.

6. OUTLET VALVE CONNECTION: (6.1) The cylinders shall be equipped with outlet valve connections designed to permit a maximum continuous withdrawal rate of 350 std cu ft per hr, and a maximum intermittent withdrawal rate of 1000 std cu ft per hr. Intermittent withdrawal shall be 5 minutes followed by a 10 minute recovery period.
(6.2) The outlet valve connections shall conform to Connection 580 of ASA B57.1.

MARKING

7. MARKING: (7.1)Each cylinder shall be marked in accordance with ASA Z 48.1.
(7.2) Each cylinder shall bear a tag plainly marked as follows: Purchase Order Number; Liquid Argon; P D Spec 52118BA (Sub A); Volume; and Name of Manufacturer.

Westinghouse Electric Corporation

Nov 5, 1966

File No.
P D Spec 52101AA thru AD Rev A

(Federal CODE IDENT NO. 79500)

HYDROGEN

1. This specification covers compressed hydrogen gas.

P D Spec Designation	Previous P D Spec	Description
52101AA	3057-1	Standard commercial grade hydrogen.
52101AB	3057-2	High purity hydrogen, low in oxygen content.
52101AC	3057-3	100% hydrogen. Purchased by brand.
52101AD	3057-4	99.9% hydrogen. Purchased by brand.

NOTE: UNLESS OTHERWISE SPECIFIED, THE FOLLOWING REQUIREMENTS APPLY ONLY TO 52101AA,AB.

2. No change shall be made in the quality of successive shipments of material furnished under this specification without first obtaining the approval of the purchaser.

MANUFACTURE

3. MANUFACTURE: The hydrogen gas shall be manufactured by the electrolytic process.

CHEMICAL PROPERTIES

4. PURITY: (4.1) (52101AA) The gas shall not contain less than 99.6 per cent hydrogen.
(4.2) (52101AB) The gas shall not contain less than 99.8 per cent hydrogen.

5. OXYGEN CONTENT: (5.1) (52101AA) Shall not be more than 0.40 per cent (4000 ppm) by volume.
(5.2) (52101AA) Shall not be more than 0.18 per cent (1800 ppm) by volume.

6. NITROGEN CONTENT: (52101AB) Shall not be more than 0.02 per cent (200 ppm) by volume.

7. MOISTURE: (7.1) (52101AA) The gas shall show no evidence of moisture when determined by inverting the cylinder and "cracking" the outlet valve.
(7.2) (52101AB) The gas shall have a dew point not higher than -47.45 C (-55 F).

PHYSICAL PROPERTIES

8. OUTLET VALVE CONNECTION: Steel cylinders in which the gas is shipped and stored shall be equipped with outlet valve connection conforming to ASA B57.1 Connection 350.

9. CYLINDERS: (9.1) Cylinders shall meet requirements of the Bureau of Explosives and Rulings of I C C (Specification 3-A).
(9.2) All cylinders shall be thoroughly purged of water or other impurities.

PACKING AND MARKING

10. PACKING: The hydrogen shall be shipped in steel cylinders holding 200 cubic feet at a pressure of 2000 pounds per square inch at a temperature of 21 C (70 F).

11. MARKING: (11.1) Each cylinder shall be plainly marked with the name of the gas in the cylinder in accordance with ASA Z48.1.
(11.2) Each cylinder shall bear a tag plainly marked as follows: Purchase Order Number; Hydrogen, stating P D Spec Number and Rev Letter; Number of cubic feet; Name of Manufacturer.

INFORMATION CAT[illegible]ORY

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Au[illegible]ized Classifier Date

Westinghouse Electric Corporation

AstroNuclear Laboratory
P. O. Box 10864
Pittsburgh, Pa. 15236
(Fed. Ident. Code No. 14683)

PURCHASING DEPARTMENT SPECIFICATION 30130
(Not for Publication)

July 10, 1968

METHANE, HIGH PURITY

1. SCOPE

This specification covers the requirements for high purity methane, designated as follows:

PD Spec Designation	Description
30130-1	Methane gas with a minimum content of 99.95 per cent methane.

2. APPLICABLE DOCUMENTS

2.1 The following documents, of the issue in effect on the date of invitation for bids, shall form a part of this specification to the extent specified herein.

USAS B 57.1

2.2 Copies of USA Standards should be obtained from the United States of America Standards Institute, 10 East 40th Street, New York, New York 10016.

3. REQUIREMENTS

3.1 CHEMICAL COMPOSITION: The gas shall conform to the following composition limits:

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GDR/as

W form 5950AA

Component	Limit or Range
Methane	99.95 percent min.
Carbon dioxide	50 ppm max.
Ethane	30 ppm max.
Nitrogen	50 ppm max.
Oxygen	10 ppm max.
Propane	5 ppm max.
Water	5 ppm max.

3.2 Containers

3.2.1 Cylinders: The cylinders shall conform to the requirements of the Bureau of Explosives and rulings of the ICC.

3.2.2 Valves: The outlet valve connection shall conform to CGA Connection 350 as defined in USAS B 57.1.

3.2.3 Marking

3.2.3.1 Each cylinder shall be legibly marked with the name of the gas. Marking shall be by means of stenciling, stamping, or labeling and shall not readily be removable.

3.2.3.2 The marking shall be located at the valve end and off the cylindrical part of the body.

3.2.3.3 The height of the lettering shall not be less than one twenty-fifth (1/25) of the diameter of the cylinder with a minimum height of 1/8 inch.

3.2.3.4 Each cylinder shall carry the name of the owner or ownership symbol, serial number, test dates, and ICC service pressure marking.

4. QUALITY ASSURANCE

4.1 Source Surveillance

4.1.1 Material covered by this specification is subject to source surveillance by a Westinghouse representative. This may include surveillance of the product and of the seller's systems, procedures, and facilities which relate to testing and inspection of this material. The seller shall perform tests and supply data as required to illustrate compliance with all requirements of this specification.

4.1.2 Material accepted by the purchaser's representative at the place of manufacture which is later found to be not in conformance with this specification is subject to rejection.

4.2 CONSISTENT QUALITY: No change shall be made in the quality of successive shipments of material furnished under this specification without first obtaining the approval of the purchaser.

4.3 PURCHASER'S INSPECTION: Cylinders shall be subject to inspection for conformance to Sections 3.1, 3.2.1 through 3.2.3.4, and 5.2.

5. PREPARATION FOR SHIPMENT

5.1 SHIPPING: The seller shall load each cylinder on the vehicle used for transportation so that it is protected from damage during shipment.

5.2 Each cylinder shall bear a tag plainly marked as follows: Purchase Order Number; Methane, PD Spec Number and Revision Letter specified on Purchase Order; Number of Cubic Feet; Name of Manufacturer.

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Westinghouse Electric Corporation

Astronuclear Laboratory
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(Fed. Ident. Code No. 14683)

PURCHASING DEPARTMENT SPECIFICATION 30177 August 17, 1965
(Not for Publication)

POWDER, ALLOY-BRAZING

1. SCOPE

This specification covers requirements for a brazing alloy powder to be used as filler material in brazing of graphite components, designated as follows:

P D Spec Designation	Description
30177-1	Titanium, zirconium, beryllium brazing alloy, 100-mesh powder

2. SUPPLIER

The supplier for 30177-1 is: Brush Beryllium Company
17876 St. Clair Avenue
Cleveland, Ohio

3. CONSISTENT QUALITY

The manufacturer shall submit a certified statement that no change has been made in the product or in the manufacture of the product specified in Section 1 since January 1, 1965.

4. LOT IDENTIFICATION

The supplier shall mark each container with an identification of the lot or batch of material and the date of manufacture.

5. STORAGE

The shelf life of the powder, stored at room temperature in sealed containers, is indefinite.

6. DESCRIPTION

The brazing alloy shall be furnished as 100-mesh powder and have the following nominal chemical composition: 48% titanium, 48% zirconium and 4% beryllium.

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